RCRA FACILITY INVESTIGATION PROPOSAL
CIBA-GEIGY FACILITY
CRANSTON, RHODE ISLAND

VOLUME 6 OF 6
ANALYTICAL SERVICES QUALITY ASSURANCE MANUAL

Submitted by:

CIBA-GEIGY CORPORATION
444 SAWMILL RIVER ROAD
ARDSLEY, NEW YORK 10502

Date:
SEPTEMBER 1989

RCRA FACILITY INVESTIGATION PROPOSAL
TABLE OF CONTENTS

VOLUME 1
INVESTIGATION WORK PLAN

VOLUME 2
CURRENT ASSESSMENT SUMMARY REPORT

VOLUME 3
PROJECT QUALITY ASSURANCE PLAN

VOLUME 4
HEALTH AND SAFETY GUIDELINES

VOLUME 5
PUBLIC HEALTH AND ENVIRONMENTAL RISK EVALUATION,
MEDIA PROTECTION STANDARDS AND CORRECTIVE
MEASURES RISK EVALUATION WORK PLAN

VOLUME 6
ANALYTICAL SERVICES QUALITY ASSURANCE MANUAL

INTERNATIONAL TECHNOLOGY CORPORATION

IT ANALYTICAL SERVICES
5815 Middlebrook Pike
Knoxville, TN

U.S. EPA CONTRACT LABORATORY PROGRAM

STANDARD OPERATING PROCEDURES (SOPs)

TO BE UTILIZED FOR THE ANALYSIS OF

APPENDIX IX COMPOUNDS

Regional Office
5815 Middlebrook Pike • Knoxville, Tennessee 37921 • 615-588-6401

TABLE OF CONTENTS

TABLE OF CONTENTS
(continued)

STANDARD OPERATING PROCEDURE

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: Receipt and Logging in of EPA Contract Laboratory Program Samples	SOP NO: CD-841010R1 DATE INITIATED: 10/10/84 REVISION NO: 1 DATE REVISED: 02/12/87 PAGE 1 of 4

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
J.M. Jones	Virginia K. Moore	2/5/87	Janet M. Jones	2/13/87

1.0 Sample Receipt

1.1 Samples Received on Weekends

1.1.1 After notification from the Sample Management Office (SMO) that samples are to be received on a weekend, the ITAS CLP Project Manager assigns weekend sample receipt to a specific lab employee.

1.1.2 Upon delivery of samples, the designated lab employee must

1.1.2.1 Sign for shipment after verifying that the number of packages received agrees with airbill/waybill.

1.1.2.2 Fill in date/time samples received on Sample Receipt Log (Figure 1).

1.1.2.3 Check ice chests for the presence and condition of Custody Seals. A Custody Seal should be positioned so that it would have to be cut, broken, or somehow disrupted for the ice chest to be opened. The condition of the Custody Seal, intact or disrupted, is noted on the Sample Receipt Log.

1.1.2.4 Move ice chest(s) to the refrigerated EPA-CLP sample storage area. Sign and date the Sample Receipt Log-relinquishing chain of custody to storage.

1.1.2.5 Place shipping papers and the sample Receipt Log in Coding Room.

1.1.3 The following Monday (or next regular work day), coding personnel must

1.1.3.1 Remove ice chests from refrigerated storage and move to the Coding Room, documenting chain of custody on Sample Receipt Log.

1.1.3.2 Review existing sample receipt information and contact the SMO if Custody Seals have been disrupted.

1.2 Samples Received on Weekdays

1.2.1 Coding personnel are notified as soon as sample shipments arrive and must

1.2.1.1 Sign for shipment after verifying that the number of packages received agrees with airbill/waybill.

1.2.1.2 Fill in date/time samples received on Sample Receipt Log (Figure 1).

1.2.1.3 Check ice chests for the presence, position, and condition of Custody Seals. A Custody Seal should be positioned so that it would have to be cut, broken, or somehow disrupted for the ice chest to be opened. The condition of the Custody Seal, intact or disrupted, is noted on the Sample Receipt Log. If Seal has been disrupted, notify the SMO before proceeding with coding.

1.2.1.4 Move ice chest(s) and shipping papers to the Coding Room.

2.0 Project/Sample Log In

2.1 Samples received on a given day are grouped together by EPA Case No., under an ITAS Project Code consisting of the 4-letter client code EPAL and a 5-digit number. The 4-letter client code is client specific and the 5-digit number is sequentially assigned as Projects are coded in. Both the Project Code and the EPA Case No. are entered on the Sample Receipt Log.

2.2 Ice chests are opened under a vented hood immediately and samples inspected as to sample condition and presence/condition of Custody Seals. This information is then entered on the Sample Receipt Log and the accompanying EPA Organics Traffic Reports. The coding personnel signs each Organics Traffic Report and enters the date sample was received.

2.3 Each sample in a Project is given an ITAS sample number consisting of a one-letter prefix and a four-digit number. Pre-numbered label tape is affixed to each sample container. These numbers are sequentially assigned as they are coded in. Samples are processed through the laboratory by sample number. The following sample information is entered on the Sample Receipt Log:

- -EPA Sample No.
- -Sample Type (from Traffic Report)
- -Sample Concentration (from Traffic Report)
- -Extract Tag No.
- -ITAS Extract Sample No.
- -Where Extract Sample Stored
- -VOA Tag No.
- -ITAS VOA Sample No.
- -Where VOA Sample Stored
- -VOA Condition (no. of vials received/no. of vials without air bubbles)
- -If sample information does not agree between Traffic Reports, Sample Tags, and Chain of Custody forms, the "NA" column is checked and the SMO office contacted. Details on discrepancies are entered in the "Notes" column.

2.4 Samples are then stored under refrigeration in secured refrigerator space designated for CLP samples only. The Sample Receipt Log is signed and dated--relinquishing chain of custody to storage.

2.5 Project/Sample information is then entered into the ITAS customized Perkin-Elmer LIMS 2000 computer data base. After the Project is entered into the computer, a Project/Sample information output is printed (Figure 2).

2.5.1 The following information is entered for the PROJECT CODE items:

Item 2-DUE DATE: (30 days from receipt of samples)

Item 4-CHAIN OF CUSTODY?: (Y)

Item 7-SPECIAL QC?: (Y)

Item 8-CLIENT TYPE (C,G,I)?: (G)

Item 15-PURCHASE ORDER NO.: (Enter EPA Case No.)

Item 16-CONTRACT NO.: (Enter EPA Contract No.)

Item 18-JOB CODE: (EPAL001)

Items 31,32,33-NOTES: (Use the following test assignments for CLP samples:

?

2.5.2 Answer "Y" to PRL prompt, "Activate Data Entry Into Sort Fields (Y/N)?".

2.5.3 The following information is entered for SAMPLE items:

Item 1-SAMPLE TYPE: (Determine appropriate ITAS code from Organics Traffic Report items 3, Sample Matrix, and 8, Sample Description)

Item 3-SPEC SAMPLE DISPOSAL: (Enter "Do not dispose of sample without authorization from ITAS CLP Project Manager")

Item 4-NOTES: (Enter "low concentration" or "medium concentration", as checked in Item 2 of the Organics Traffic Report)

Item 8-SAMPLE DESCRIPTION: (Enter the description found in the first column of Item 6 on the Organics Traffic Report, i.e. "Water (Ext.)" or "Water (VOA)", etc.

Item 12-CLIENT SAMPLE NO.: (Enter "EPA Case No.-EPA Sample No.", i.e. "7740-C4401")

Item 13-SORT CODE 1: (Enter EPA Case No.)

Item 14-SORT CODE 2: (Enter EPA Sample No.)

Item 22-FIELD SAMPLE NO.: (Enter EPA Tag No.)

2.6 Each Project is assigned a file folder labeled with the Project Code and EPA Case No. All coding information printouts, Chain of Custody forms, Traffic Reports, Sample Receipt Logs, EPA Sample Tags, and shipping papers are filed in the Project Folder. These Project Folders are then filed in the in-coming Project box in the room set aside for EPA-CLP document storage and administrative activities.

(ITAS-K-CD001RO)

SAMPLE RECEIPT LOG
EPA CONTRACTUAL SAMPLES
ITAS-Knoxville

Project Code _____
Contract # _____
SMO Case # _____

Date/Time Received _____
Required Turnaround _____

Sample Condition
Cold Yes__ No__
Intact Yes__ No__

Custody Seals	On Ice Chests	On Samples	Forms	Received	Agree*
Present	Yes___ No___	Yes__ No__	Federal Express Airbill	Yes__ No__	
Properly Placed	Yes___ No___	Yes__ No__	Traffic Report	Yes__ No__	Yes__ No__
Intact	Yes___ No___	Yes__ No__	Sample Tags	Yes__ No__	Yes__ No__
			Chain of Custody	Yes__ No__	Yes__ No__

* If No, then "x" in "NA" column below will indicate which samples are affected by Non-Agreement.

EPA Sample #	Sample Type	Sample Conc.	Extract Tag #	ITAS Extract Sample #	Where Stored	NA	VOA Tag #	ITAS VOA Sample #	Where Stored	VOA Cond.	NA	Notes

For Weekend Receipt:

Ice chests put in refrigerated storage _____ (Date) _____ (Initials)

Samples put in refrigerated storage _____ (Date)

Ice chests removed from refrigerated storage _____ (Date) _____ (Initials) _____ (Signature)

PROJECT=EPAL23446 DATE=02/12/87 TIME= 15:49:4

PROJECT & TEST ASSIGNMENT VERIFICATION

PROJECT CODE	EPAL23446
DATE-TIME ENTERED	11/19/86 17:04:18
DATE-TIME MODIFIED	02/12/87 15:41:50
STANDARD TESTS	
DATE SAMPLES REC'VD	11/19/86
DUE DATE	12/19/86
PROJ.STATUS	P
CHAIN OF CUSTODY?	Y
RUSH STATUS?	N
CALL REPORT?	N
SPECIAL QC?	
CLIENT TYPE	G
CALL REPORT TO	
BILLING CODE	
PROFIT CENTER	4620
CLIENT PO#	
PROJECT STATUS HIST.	PON444448
QUOTATION	
PURCHASE ORDER NO.	6500
CONTRACT NO.	EPA-68-01-7025
PROPOSAL NO.	
IT JOB #	EPAL001
INITIALS-QC APPROVAL	
ITAS JOB #	
INITIALS-PROJ.RELEAS	RMH
DATE REPORTED	12/16/86
REPORT FORMAT	
DATE INVOICED	12/31/86
DATE # REC'VD	
OTHER SUBMISSION INF	See C of C & traffic report
PROJ.DESCRIP1	Sixteen (16) water samples
PROJ.DESCRIP2	
SAMPLE #S ASSIGNED	AA5042\AA5058,AA5059\AA5074
SAMPLE #S ASSIGN	
NOTES #1	AA5042\AA5058 (VOAs by CLP protocol),AA5059\AA5074 (BN/AE by CLP
NOTES #2	protocol & Pest/PCBs by CLP protocol)
NOTES #3	
PROJECT NUMBER	23446
SUBPROJECT ?	N
TRACKING NET	PR
(RESERVED)	
LIMS FILE	

___MM Group ___GC/MS Group
___Metals Group ___Organic Prep. Group
___GC & Misc. Org. Groups ___Misc. Inorganic Group

SAMPLES AA5042\AA5058

TEST ! MM09 817	EPA CLP Hazardous Substance VOAs in Water	EPA CLP Organic Analysis Protocol, Contract 6
TEST ! MM09 818	EPA CLP Tentatively Identified 10 High. Add. VOAs	EPA CLP Organic Analysis Protocol, Contract 6

SAMPLES AA5059\AA5074

TEST ! ME12 817	EPA CLP Hazardous Substance BN/AEs in Water	EPA CLP Organic Analysis Protocol, Contract 6
TEST ! GC19 817	EPA CLP Hazardous Substance Pest./PCBs in Water	EPA CLP Organic Analysis Protocol--Contract 6
TEST ! ME12 819	EPA CLP Tentatively Identified 20 High. Add. BN/AE	EPA CLP Organic Analysis Protocol, Contract 6
TEST ! PM12	EPA CLP Hazardous Substance BN/AEs in Water	EPA CLP Organic Analysis Protocol, Contract 6
TEST ! P619	EPA CLP Hazardous Substance Pest./PCBs in Water	EPA CLP Organic Analysis Protocol, Contract 6

••
••

AA5042 7025-88583 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)

PROJECT=EPAL23446 DATE=02/12/87 TIME= 15:49:4

NOTES : Low Concentration
AA5043 7025-BF860 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5044 7025-BF861 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5045 7025-BF862 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5046 7025-BF869 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5047 7025-BF871 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5048 7025-BF872 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5049 7025-BF874 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5050 7025-BF875 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5051 7025-BF876 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5052 7025-BF877 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5053 7025-BF878 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5054 7025-BF879 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5055 7025-B6886 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5056 7025-B6338 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5057 7025-BF862 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration
AA5058 7025-BF862 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (VOA)
NOTES : Low Concentration

••

AA5059 7025-88583 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (E.t.)
NOTES : Low Concentration
AA5060 7025-BF860 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001
SAMPLE DESCRIPT. : Water (E.t.)
NOTES : Low Concentration
AA5061 7025-BF861 STAT=07 TYPE=01 TNET=PR PROJ=EPAL23446 TASA= JOB=EPAL001

STANDARD OPERATING PROCEDURE

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: Sample Storage for EPA Contract Laboratory Program	SOP NO: QA841113R1 DATE INITIATED: 11/13/84 REVISION NO: 1 DATE REVISED: 02/13/87 PAGE 1 of 2

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
James E. Atwell	Jack H Hall	2/18/87	Janet M Jones	2/18/87

1.0 Samples and extracts will be stored in secured, refrigerated areas designated for EPA-CLP samples. Separate areas will be defined for raw extract samples, VOA samples, GC/MS extracts, GC extracts, and archival of sample extracts and raw sample after completion.

1.1 Refrigerators will be kept locked at all times. The following ITAS employees will be issued keys to the refrigerated areas: Sample Custodians, EPA-CLP Project Manager, EPA-CLP Document Coordinator, GC/MS Group Leader, GC Group Leader, and Organic Prep Group Leader. The EPA-CLP Project Manager may also sign out spare keys to designated employees in order to cover weekend and shift operations.

1.2 Refrigerated areas will be designated, operated, and monitored according to contract specifications or other EPA directives. ITAS Support Services personnel are responsible for the monitoring and operation of refrigerators and refrigerated areas in accordance with SOP MA841214R0.

2.0 Whenever samples or extracts are transferred into or out of "in-process" storage, the transfer will be documented using EPA-CLP Sample Receipt Logs or the Project Work Sheets used for analysis assignments to the laboratory groups. Documentation will be in accordance with SOP CD841010R0, "Receipt and Logging in of EPA Contract Laboratory Program Samples" and SOP PM841011R0, "Work Assignments, Analysis Tracking and Sample Chain of Custody for the EPA Contract Laboratory Program".

3.0 Samples and extracts will be stored/archived after completion of analyses. Sample Archive Logs (Figure 1) will be used for documenting transfer into or out of archive storage, including disposal. These Logs will be attached to the sample archive refrigerators or refrigerated rooms. Disposal of samples will occur after one of the following:

(1) 180 days after data submission, the ITAS Project Manager will submit to the EPA Project Officer, a written request to dispose of samples. Upon written authorization back from the Project Officer, samples will be disposed.

(2) Seven days after receipt of written disposal request from the EPA Project Officer or the Sample Management Office.

3.1 Samples will be disposed of in an appropriate manner as set forth in the IT Corporation Laboratory Safety Manual.

SAMPLE ARCHIVE LOG
EPA-CLP Program

Refrigerator ID: _____ Page No. _____

Laboratory Sample No.	Date/Time Archived	Date/Time Removed	Reason (Re-analysis, Disposal, Return to EPA, etc.)	Date/Time Returned

STANDARD OPERATING PROCEDURE

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE:	SOP NO: QA841214R2-1 DATE INITIATED: 12/14/84 REVISION NO: 2 DATE REVISED: 11/06/87 PAGE 1 of 2

Work Assignments, Analysis Tracking, and Sample Chain of Custody for the EPA Contract Laboratory Program

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
[signature]	[signature]	11/12/87	[signature]	11/12/87

1.0 After projects have been coded in and samples stored under refrigeration according to SOP ITAS-K-CD841010RO, coding personnel are responsible for notifying the EPA-CLP Document Coordinator that samples have arrived.

1.1 The Document Coordinator reviews coding information from the Project Folder and verifies the test assignments. Group Supervisors verify the computer test assignments (see Figure 1) and update Project Status to "Test Assigned/Modified by GRL's". After the Project is verified, the Group Supervisors update the Project Status to "Test Assignments Verified by Coordinators".

1.2 After verification, the Group Supervisors are responsible for producing Project Work Sheets for analysis assignments to the laboratory groups. Copies of the Work Sheets for GC, GC/MS, and Organic Prep are shown in Figures 2-6.

1.3 Group Supervisors check their Work Sheets and update their Master Project Logs shown in Figures 7-9. Work assignments are distributed to group personnel by use of the Work Sheets.

1.4 The Project Work Sheets are held in the various laboratory groups until completion of the prep or analysis for all samples. They are signed and dated by analysts or prep technicians after prep or analysis has been completed and then turned in to the Group Supervisor in charge. After review, the Group Supervisor approves the work and either sends the Work Sheet to the analysis group involved (in the case of Organic Prep) or attaches the Work Sheet to the analysis forms and turns it in to the Document Coordinator (in the case of analysis groups).

1.4.1 Chain-of-Custody of samples moving through the laboratory for analysis will be documented by the use of Internal Chain-of-Custody (Figure 10). Spaces are provided for entering employee name, date, and location where samples were taken from storage, where extracts were stored, and where left over sample and completed extracts were stored.

1.5 Computer analysis status updates are entered by analysts after completion of assignments and by Group Supervisors after reviewing and approving data. Figure 11is a list of Project and Sample/ Analysis status codes.

1.5.1 Using the computer system, the status of samples can be monitored at the Project, Sample, Analysis, or Case level. Printouts can be created at any time or information viewed on CRT's.

1.6 The Document Coordinator organizes and assembles the data packages using the EPA-CLP Data Package Checklist shown in Figure 12. The data package is reviewed by both the Document Coordinator and the GC/MS Group Supervisor. The QC Coordinator reviews 5% of the data packages before release.

1.7 After approval of the data packages associated with a project code, the Document Coordinator makes copies of the QC data summaries and gives them to the Assistant Lab Manager. This indicates the data package is ready to be mailed and serves as documentation for invoicing. The computer is then updated to "Report Released (Mgt)" and "Report Has Been Sent Out" under direction of the Assistant Lab Manager.

1.8 Completed Projects are filed in the EPA-CLP room by Case No. in locked filing cabinets. All records associated with the CLP samples are stored together--shipping papers, SMO documents, coding information, Work Sheets, raw data, and the copy of the final data packages.

FIGURE 1

PROJECT & TEST ASSIGNMENT VERIFICATION

___WW Group	___GC/MS Group
___Metals Group	___Organic Prep. Group
___GC & Misc. Org. Groups	___Misc. Inorganic Group

FIGURE 2

PROJECT PREP WORKSHEET FOR GROUP LEADERS PAGE 1

ORGANIC PREP

PROJECT CODE=EPAL23725 DUE DATE=1/26/87 DATE ISSUED=01/26/87 14:52

SAMPLE(S)	TY	R?	DUE DATE	PREP	PREP DESCRIPTION
AA7216\AA7219	01	E		PG19	EPA CLP Hazardous Substance Pest./PCBs in Water

INSTRUCTIONS:

SPECIAL QC :

PREP-NOTES :

PREPPED BY: _____ / / APPROVED BY: _____ / /

FIGURE 3

PROJECT PREP WORKSHEET FOR GROUP LEADERS PAGE 1

ORGANIC PREP

PROJECT CODE=EPAL23725 DUE DATE=01/26/87 DATE ISSUED=01/26/87 14:55

SAMPLE(S)	TY	R?	DUE DATE	PREP	PREP DESCRIPTION
AA7216\AA7219	01	E		PM12	EPA CLP Hazardous Substance BN/AEs in Water

INSTRUCTIONS:

SPECIAL QC :

PREP-NOTES :

PREPPED BY: _____ / / APPROVED BY: _____ / /

FIGURE 4

PROJECT ANALYTICAL WORKSHEET FOR GROUP LEADERS PAGE 1

QC

PROJECT CODE=OLIT23788 DUE DATE=2/12/87 DATE ISSUED=02/10/87 10:24

SAMPLE(S)	TY R?	DUE DATE	TEST	TEST DESCRIPTION
AA7496	11 E		GC03 808	PCBs, as Aroclors, in Oils & Natural Gas Condensate (ASTM)

INSTRUCTIONS:

SPECIAL QC :

ANAL-NOTES :

ANALYST: _____, __/__/__ APPROVED BY: _____, __/__/__

FIGURE 5

PROJECT ANALYTICAL WORKSHEET FOR GROUP LEADERS PAGE 1

GC/MS VOA'S - CLP

PROJECT CODE=EPAL23792 DUE DATE=2/24/87 DATE ISSUED=02/11/87 20:48

SAMPLE(S)	TY	R?	DUE DATE	TEST	TEST DESCRIPTION
AA7518\AA7522	31	E		MV10 817	EPA CLP Hazardous Substance VOAs in Low Level Soil
	31	E		MV10 818	EPA CLP Tentatively Identified 10 High. Add.VOA in Low Soil
AA7523\AA7526	01	E		MV09 817	EPA CLP Hazardous Substance VOAs in Water
	01	E		MV09 818	EPA CLP Tentatively Identified 10 High. Add. VOAs in Water

INSTRUCTIONS: FIVE (5) LOW CONC. SOIL SAMPLES AND FOUR (4) WATER SAMPLES FOR VOA'S BY CLP PROTOCOL.

SPECIAL QC :

VAL-NOTES :

ANALYST: _____, __/__/__ APPROVED BY: _____, __/__/__

FIGURE 6

PROJECT ANALYTICAL WORKSHEET FOR GROUP LEADERS PAGE 1

GC/MS BN/AE'S - CLP

PROJECT CODE=EPAL23792 DUE DATE=3/10/87 DATE ISSUED=02/11/87 20:40

SAMPLE(S)	TY	R?	DUE DATE	TEST	TEST DESCRIPTION
AA7510\AA7514	31	E		ME13 817	EPA CLP Hazardous Substance BN/AEs in Low Level Soil
	31	E		ME13 819	EPA CLP Tentatively Identified 20 High.Add.BN/AE in LowSoil
AA7515\AA7517	01	E		ME12 817	EPA CLP Hazardous Substance BN/AEs in Water
	01	E		ME12 819	EPA CLP Tentatively Identified 20 High. Add. BN/AEs in H2O

INSTRUCTIONS: FIVE (5) LOW CONC. SOIL SAMPLES AND THREE (3) WATER SAMPLES FOR BN/AE'S BY CLP PROTOCOL.

SPECIAL QC :

..NAL-NOTES :

ANALYST: _____, __/__/__ APPROVED BY: _____, __/__/__

FIGURE 7

ORGANIC PREP

Project Code	Sample Type	Samples Nos.	Type of Analysis	Extracted	Concen-trated	Due Date

FIGURE 8

INTERNATIONAL TECHNOLOGY CORPORATION

By________ Date________ Subject________________________ Sheet No.____ of____

Chkd. By____ Date________ ________________________ Proj. No.________

PROJECT CODE	SAMPLE NUMBER	ANALYSIS	SAMPLE TYPE	DATE DUE	DATE REPORTED	NUMBER SAMPLES	NOTES

108-10-85

FIGURE 9

GC/MS SAMPLE RECEIPT LOG

Project Code	Sample Type	Samples Nos.	Type of Analysis				Prep Due Date	Due Date
			VOA	BN	AE	Other		

FIGURE 10

ITAS-K-QA003R2

Project Code _____
EPA Case # _____
Sample No. Range _____

INTERNAL CHAIN-OF-CUSTODY FORM - ORGANICS
IT Analytical Services-Knoxville

Sample No.	Date	Extract* Type or Original Sample?	Moved From	Moved To	Reason	Signature
		-				

* O - Original Sample
A - Acid Extract
P - Pesticide/PCB Extract
E - BN/AE Extract
B - Base Neutral Extract
V - VOA Sample

PROJECT STATUS

A	Samples Logged-In
B	Samples Logged-In & All Tests Assigned @ A
C	Test Assignments Completed by GRLs or Coordinators
D	Test Assignments Verified by Coordinators
E	On Hold
F	Help Requested
G	In-Process
H	Re-Work
I	Lab Work Completed
J	Lab Work Release In-Process
K	All Lab Work Released
L	QC Approved
M	Report Released (Mgt.)
N	Report Called to Client
O	Report Was Been Sent Out
P	Billed
Q	Partially Paid
R	Full Payment Received
S	Uncollectable
T	Cancelled
U	No Charge Project
Y	Special Long-Term, On-Going Projects--Will Have Sub-Projects
Z	Special Projects Which Will Never Have Samples

1	Samples Added before Status C
2	All Tests Assigned by Coding after Project Log-In (PRL)
3	Partial Test Assignment at Coding
4	Tests Assigned/Modified by GRLs
5	Samples Added after Status of D
6	Tests Added after Status of D
7	Samples Deleted after Status of D
8	Tests Deleted after Status of D
9	Not Released from Coding(Problem)

SAMPLE STATUS

1	Logged In
2	On Hold
3	Rejected
4	Help Requested
5	In Process
6	Re-Work
7	Complete
8	Released
9	Cancelled
10	Active (1-7 above)

FIGURE 11

TABLE I

ORGANICS

Each file must contain the following documents or a memo explaining their absence (one memo may cover several documents):

- File inventory
- * Chain-of-custody form
- * Sample tag(s)
- * Airbill(s)
- * Organics Traffic Report(s)
- * Organics Laboratory Chronicles (Extraction)
- * Organics Laboratory Chronicles (Analysis)
- Organics Analysis Data Summaries
- Copies of analyst's notebook pages
- Benchsheets and worksheets
- Copies of instrument logbook pages
- Sample tracking documents
  - Sample receipt logbook pages
  - ** Internal custody records
- Hard copies of mass spectra and chromatograms
- QA/QC package
  - DFTPP/BFB calibration spectra and worksheets
  - Quality Control Reports
  - Standards Analysis Report forms, worksheets, and Spectra/ Chromatograms
  - Duplicate, Matrix, Surrogate Spike Results
- GC/MS computer library search worksheets and accompanying spectra
- Related correspondence and/or memos
- All other related documents

* If received with sample shipment

** If used to supplement sample tracking system

FIGURE 12

STANDARD OPERATING PROCEDURE

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: Temperature Monitoring of Refrigerated Sample Storage Areas	SOP NO: MA841214R1 DATE INITIATED: 12/14/84 REVISION NO: 1 DATE REVISED: 02/13/87 PAGE 1 of 2

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
JM Jones	Clyde K. Moore	2/13/87	Janet M. Jones	2/13/87

1.0 Purpose and Applications

1.1 The purpose of this procedure is to ensure compliance with the CLP contract requirements regarding refrigerated sample storage.

1.2 This procedure applies to all refrigerated areas designated for storage of CLP samples and extracts.

2.0 Procedure Mechanically Driven Temperature Recorders

2.1 Mechanically driven temperature recorders shall be installed in all such refrigerated areas.

2.2 Such recorders shall be capable of continuously recording the temperature for not less than seven (7) days.

2.3 Each Wednesday, a designated person shall change the charts, dating and initialling them when they are installed and removed.

2.4 Completed charts shall be delivered to the Document Control Coordinator for filing.

2.5 Deviations from the contract stipulated temperature (4°C) shall be immediately reported to the QC Coordinator and appropriate corrective action taken.

3.0 Procedure Thermometers

3.1 In all refrigerators where recording thermometers are not present, a manual thermometer will be installed.

3.0 Procedure Thermometers (continued)

3.2 Every morning these thermometers will be read and the temperature recorded in a designated logbook. See Figure 1 for an example logbook page.

3.3 This logbook will be in the possession of the Coding Specialist and will be available for inspection as necessary.

3.4 Deviations from 4°C shall be immediately reported to the QC Coordinator and appropriate corrective action taken.

PROJECT _____ Continued From Page _____

Refrigerator #	Date	Time	Temp.
20	2/3/87	8:25 AM	5°C
2	"	"	3.5°C
3	"	"	5°C
4	"	"	42°F
1	"	8:30 AM	5°C
6	"	"	3.5°C
13	"	"	1°C
14	"	"	4°C
19	"	"	3°C
7	"	12:38 PM	37°F
5	"	"	2°C

Refrigerator #	Date	Time	Temp.
20	2/4/87	8:50 AM	4°C
2	"	"	4°C
3	"	"	6.5°C
4	"	8:52 AM	46°F
1	"	"	5°C
6	"	8:53 AM	5°C
13	"	8:54 AM	1°C
14	"	" AFTER	2°C
19	"	8:55 AM	4°C
7	"	"	34°F
5	"	"	2°C

Continued on Page

Read and Understood By

Signed	Date	Signed	Date

STANDARD OPERATING PROCEDURE

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: Document Numbering and Inventory Procedure for Use in EPA Contract Laboratory Program	SOP NO: QA841213R2 DATE INITIATED: 12/13/84 REVISION NO: 2 DATE REVISED: 10/30/87 PAGE 1 of 3

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Paula McElley	Jack L Hall	11/17/87	Mary E Taylor	11/13/87

1.0 Document Numbering

1.1 Document is defined for file purge purposes as any item associated with an EPA CLP case which will receive a unique document number.

1.2 The document coordinator will assign a document inventory number to each document of a case. The document number will consist of:

1.2.1 SMO case number
1.2.2 EPA region number
1.2.3 Serialized document number

1.3 All documents pertaining to each case will be assembled in the following order: (Documents 1.3.4 - 1.3.7 are divided by fraction. See Figure 1 for those files based on the 7/87 SOW.)

1.3.1 Document file inventory
1.3.2 Sample data summary
1.3.3 Case narrative
1.3.4 QC summary
1.3.5 Sample data package
1.3.6 Standards data package
1.3.7 Raw QC data package
1.3.8 Sample shipment and custody information

1.4 The sample shipment and custody information (see 1.3.8) portion will contain all the data and materials not previously submitted to the EPA for a particular case. These shall be grouped according to analytical method and document type. The documents to be included, and their order, will be as follows:

1.0 Document Numbering (continued)

1.4.1 Sample receipt log

1.4.2 Internal Chain-of-Custody records

1.4.3 Prep

1.4.3.1 GC prep worksheet and benchsheet

1.4.3.2 GC/MS prep worksheet and benchsheet

1.4.3.3 Prep screen data

1.4.3.4 pH analysis

1.4.4 GC/MS

1.4.4.1 GC/MS analytical worksheet

1.4.4.2 VOA holding blank

1.4.4.3 GC/MS instrument logs

1.4.5 GC

1.4.5.1 GC analytical worksheet

1.4.5.2 GC raw data

1.4.5.3 GC chromatograms

1.4.5.4 GC/MS pesticide confirmation

1.4.5.5 GC instrument logs

1.4.6 Related documents

1.4.6.1 Project/coding sheets

1.4.6.2 Organics traffic reports

1.4.6.3 Data receipt acknowledgements

1.4.6.4 Lab to receipt airbills (data submission)

1.4.6.5 External Chain-of-Custody

1.4.6.6 To lab airbill (sample shipment)

1.0 Document Numbering (continued)

1.4.6.7 Phone log

1.4.6.8 Sample tags

1.4.6.9 Request for Analysis

1.4.7 Contract compliance screen

1.4.7.1 Additional data

1.4.7.2 Miscellaneous

1.4.7.3 Data release

1.5 If an item that should be in the file is missing, it should be replaced by a note (written on paper containing the IT letterhead) which explains the item's absence. This should be signed and dated by the Document Coordinator.

2.0 Document Inventory

2.1 A document file inventory which includes all documents, their document file number, and total pages will be prepared and filed with each case. (See Figures 1 or 2.)

2.2 All case documents will be submitted either within seven (7) days of receipt of written request from the P.O. or SMO, or 180 days from the date of data submission. The lab will retain a copy of the document file inventory for each case submitted.

3.0 Case File Assembly

3.1 All documents will be compiled in case file folders for submission to the EPA.

3.2 Using appropriate file folders, assign one folder to each case according to SMO case number.

3.3 Place all the documents and laboratory generated data pertaining to one case in the folder.

3.4 Documents should be arranged by document type within the folder.

3.5 The document case files will be stored in secured file cabinets and arranged by SMO case number.

FIGURE 1
(1987 SOW)

Document File Inventory

Document Control Number	Document	No. of Pages
- -001	Document File Inventory	
- -002	Sample Traffic Reports	
- -003	Sample Data Summary	
- -004	Case Narrative	
- -005	VOA QC Summary	
- -006	VOA Sample Data Package	
- -007	VOA Standards Data Package	
- -008	VOA Raw QC Data Package	
- -009	BNA QC Summary	
- -010	BNA Sample Data Package	
- -011	BNA Standards Data Package	
- -012	BNA Raw QC Data Package	
- -013	PEST/PCB QC Summary	
- -014	PEST/PCB Sample Data Package	
- -015	PEST/PCB Standards Data Package	
- -016	PEST/PCB Raw QC Data Package	
- -017	Sample Receipt Log	
- -018	Internal Chain of Custody Forms	
- -019	GC Prep Worksheet	
- -020	GC Prep Benchsheet	
- -021	GC/MS Prep Worksheet	
- -022	GC/MS Prep Benchsheet	
- -023	Prep Screen Data	
- -024	pH Analysis	
- -025	GC/MS Analytical Worksheet	
- -026	VOA Holding Blank	
- -027	GC/MS Instrument Logs	
- -028	GC Analytical Worksheet	
- -029	GC Raw Data	
- -030	GC Chromatograms	
- -031	GC/MS Pesticide Confirmation	
- -032	GC Instrument Logs	
- -033	Project/Coding Sheets	
- -034	Data Receipt Acknowledgements	
- -035	Lab to Receipt Airbills (Data Submission)	
- -036	External Chain of Custody	
- -037	To Lab Airbill (Sample Shipment)	
- -038	Phone Log	
- -039	Sample Tags	
- -040	Request for Analysis	
- -041	Contract Compliance Screen	
- -042	Additional Data	
- -043	Miscellaneous	
- -044	Data Release	
	Total Number of Pages	_____

FIGURE 2
(1985 SOW)

Document File Inventory

Document Control Number		Document	No. of Pages
-	-001	Document File Inventory	
-	-002	Sample Data Summary	
-	-003	Case Narrative	
-	-004	QC Summary	
-	-005	Sample Data Package	
-	-006	Standards Data Package	
-	-007	Raw QC Data Package	
-	-008	Sample Receipt Log	
-	-009	Internal Chain of Custody Forms	
-	-010	GC Prep Worksheet	
-	-011	GC Prep Benchsheet	
-	-012	GC/MS Prep Worksheet	
-	-013	GC/MS Prep Benchsheet	
-	-014	Prep Screen Data	
-	-015	pH Analysis	
-	-016	GC/MS Analytical Worksheet	
-	-017	VOA Holding Blank	
-	-018	GC/MS Instrument Logs	
-	-019	GC Analytical Worksheet	
-	-020	GC Raw Data	
-	-021	GC Chromatograms	
-	-022	GC/MS Pesticide Confirmation	
-	-023	GC Instrument Logs	
-	-024	Project/Coding Sheets	
-	-025	Organics Traffic Reports	
-	-026	Data Receipt Acknowledgments	
-	-027	Lab to Receipt Airbills (Data Submission)	
-	-028	External Chain of Custody	
-	-029	To Lab Airbill (Sample Shipment)	
-	-030	Phone Log	
-	-031	Sample Tags	
-	-032	Request for Analysis	
-	-033	Contract Compliance Screen	
-	-034	Additional Data	
-	-035	Miscellaneous	
-	-036	Data Release	
	—		
		Total Number of Pages	_____

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE:	SOP NO: QA851023R1 DATE INITIATED: 10/23/85 REVISION NO: 1 DATE REVISED: 02/13/87 PAGE 1 of 1
Quality Control Practices, Data Validation, and Acceptability Criteria for Samples Analyzed by USEPA Contract Laboratory Program Protocol	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Gary M Woody	Jack M Hall	2/18/87	Janet M Jones	2/18/87

1.0 Scope and Applications

This procedure applies to all aqueous and solid samples submitted to ITAS for analysis for Hazardous Substance List (HSL) compounds following procedures specified in EPA IFB's WA-85J664 (organics) and WA-85J839 (inorganics) and subsequent amendments.

2.0 Quality Control Practices

2.1 Analytical methods and instrument calibrations shall be as specified in Exhibit D of the respective IFB's.

2.2 Frequency of calibration checks and QC samples (e.g., method blanks, duplicate samples, spiked samples, etc.) shall be as specified in Exhibit E of the respective IFB's.

3.0 Data Validation

As is appropriate, the Assistant Laboratory Manager or the QC Coordinator will review the data for correctness of calculations and data transcription, proper reporting units, QC requirements, and completeness of data and deliverables. Any qualification of the data will be made by the above persons following procedures specified in Exhibit B of the respective IFB's. Only after such validation will the Assistant Laboratory Manager, or his designate, approve the data for release to the client.

4.0 Acceptability Criteria

Acceptability of the data shall be determined by criteria specified in Exhibit E of the respective IFB's. Such criteria include, but are not limited to, calibration verifications, GC/MS tunes, GC retention times, relative percent differences at duplicates, spike recoveries, and method blanks.

STANDARD OPERATING PROCEDURE

INTERNATIONAL TECHNOLOGY CORPORATION

ITAS-KNOXVILLE

TITLE:	SOP NO: AP871106R0
Standardization for Analysis by Inductively Coupled Plasma Emission Spectroscopy by CLP Protocol	DATE INITIATED: 11/06/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 8

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Janet M Jones	Jackie Hall	11/12/87	Mary E Tyler	11/12/87

1.0 Purpose

This method describes a technique for simultaneous multielement determination of trace elements in solution. Refer to SOP A_871105R0 and A_871106R0 for details on sample preparation.

This procedure describes instrument operation, standardization, sample analysis and quality control guidelines in accordance with CLP SOW 787.

2.0 Starting the Instrument

Unless the instrument will not be used for a long period of time, leave the main power on. Refer to the operator's manual for considerations when performing a cold start.

2.1 Turn on the water recirculator and open the main valve to the argon tank. Flip the toggle switches for torch flow and sample flow, which are on the front right side of the instrument, to the on position. With the torch chamber door (front of instrument) closed, allow the system to purge for five minutes. Measure argon flow from bottom of flow bead. The torch flow should be close to 15. During the last minute or so, operate the peristaltic pump and observe the nebulizer spray chamber. The absence of a fine mist indicates a clogged capillary. While the torch is being purged, the computer system can be started.

2.2 Turn on the printer, terminal, and computer. Follow the instructions which appear on the terminal. When the "$" appears as a prompt, log on by typing the following:

2.2.1 HELLO
1,11 (in response to question for account #)
TEJA (password)

2.0 Starting the Instrument (continued)

2.2.2 You will be logging onto the "A-to-Z" system. When the menus appear, choose SA, then OI, and then the sample analysis program, SAT. You may indicate the desired menu item by placing the cursor (on the keyboard) or by typing in the abbreviation for the item. Press the "return" key after the choice is made.

2.2.3 Once into the SAT program, you will be prompted for the name of an ACT (Analytical Control Table). This table contains all of the information needed for analysis of a particular group of metals. Standardization values are stored there as they are updated. (Note that the term standardization is used instead of calibration, as the section in the operator's manual which is labeled "calibration" is aimed specifically at solid sampling instruments.)

2.2.4 Refer to the operator's manual for instructions on how to create an ACT. Type in the name of the ACT that you wish to use and press the "return" key.

2.2.5 An indirect command file has been installed which will guide you through profiling the instrument and standardization. After standardization, the command file will set a command string to perform three burns. The command file is activated by typing @ CLP for the CLP ACT. (@ FULL for the FULL ACT and @ CORN for the CORN ACT.)

2.3 Before initiating the plasma, verify that the instrument setup is as follows:

2.3.1 R.F. off button is lit (blue). If not, verify that water recirculator has been turned on and that plasma chamber door on front of instrument is closed. Note: If this door is opened during analysis, the plasma will go out.

2.3.2 The automatic power control switch is in manual position.

2.3.3 The power control knob is fully counterclockwise.

2.3.4 The load control tuning switch is in automatic position.

2.3.5 "SB" and "FAT" indicators on the controller panel should be lit (red). If the instrument is on and these indicators are not lit, push the reset button on the controller panel. If this fails, push the spectrometer reset button on the back of the instrument. Refer to the operator's manual for a discussion of when this may be necessary.

2.0 Starting the Instrument (continued)

2.4 Profiling, centering the spectral lines on their respective exit slits, should be performed at the start of each day. If the mercury pen lamp is not used, you must profile on the spectral line used when the instrument was prepared by the manufacturer. As only one spectral line is profiled, all others are preset relative to the profiled line. This line is specified in the instrument logbook. See pages 6-3 and 6-4 in the operator's manual for profiling instructions. Use the mercury pen lamp.

2.4.1 After initiating the command file in Section 2.2.5, the CRT will prompt to profile the instrument. When profiling is complete, enter "0".

2.5 After profiling, you are ready to initiate the plasma.

2.5.1 Turn off the peristaltic pump. Lower the sample flow, by turning the front panel control knob, to zero. Leave the sample flow toggle switch open.

2.5.2 Push the button labeled "R.F." on. It should light immediately (red).

2.5.3 Locate the ignitor button on the front lower right side of the instrument. It is underneath the torch chamber. Stoop just enough so that you can barely see the streamers of the plasma when initiation begins. This will help you to start the plasma. Eventually, you will be able to judge plasma start-up by listening only. Please note that the door on the left side of the instrument and torch chamber does not have an interlock. It is possible to open it while the plasma is up so that viewing height can be adjusted. NEVER LOOK DIRECTLY AT THE PLASMA, as serious eye damage can result. View it only through the front panel viewing window or by looking at the image on the torch height grid.

2.5.4 Locate the power control knob and turn it clockwise until the forward power meter shows ~ 0.5 kw. Do not turn higher than ~ 0.6 kw for this particular step. Tap the ignitor button quickly and release. You may need to do this a few times. DO NOT hold the ignitor button down. Successful ignition will produce faint blue streamers which remain after the button is released. Depending on the torch position in the coil, this step may be slightly difficult.

2.0 Starting the Instrument (continued)

2.5.5 After the streamers appear, turn the power control knobs slowly up to ~ 1 kw where you will note that the plasma is trying to come up. Do not go beyond 1.1 kw. The process is somewhat noisy, so do not be alarmed. The plasma should come up at this point with the disappearance of the streamers and the appearance of the characteristic plasma glow. As mentioned earlier, work toward being able to initiate the plasma without looking at the torch. You may be able to use the viewing window instead of stooping.

2.5.6 If the plasma fails to light, an alarm (for reflected power) may go off. Simply push the reset button on the top control panel in the middle of the instrument. You do not need to push the O.L reset button on the middle panel unless the upper one does not work. Turn the power control knobs fully counter-clockwise and start over at Step 2.5.3.

2.5.7 When the plasma comes up, flip the automatic power control switch to the automatic position. Power to the torch will now be controlled by the rheostat located to the right of the power control switch. Refer to the operator's manual for a discussion of how to change this setting.

2.5.8 Turn the power control knob (same one as used to initiate the plasma) fully clockwise. The forward power should be at 1.1 kw. Maintain this setting for all aqueous samples. Before attempting to analyze organic samples, refer to the operator's manual. Turn on the peristaltic pump. Slowly turn the sample flow up to 0.65 on the flow meter. Note the "tunnel" appearing in the center of the plasma which indicates that a sample is being introduced.

2.5.9 Allow the plasma to stabilize for approximately thirty minutes before beginning standardization.

3.0 Standardization

3.1 Begin by typing the command "IS" for initiate standardization on the keyboard. (If needed, refresh screen by pushing the space bar or the "refresh screen" key at top of keyboard.) Push the "return" key. A series of standard names taken from the ACT will then be displayed. These are the standards that you must use for standardization. Their composition is specified in the ACT that you are working with.

3.0 Standardization (continued)

3.2 Aspirate the first standard. Allow at least sixty seconds for equilibration before analyzing. The following string of commands is a typical one for analyzing a sample and would be typed as it appears with the exception of the blocks used here for identification:

EA	AN	TY	ANTYANTYANTY	TA	PA
#1	#2	#3		#4	#5

3.2.1 This command string says erase previous burn from buffer (#1 - this command must preface every analysis command sequence).

3.2.2 Perform four exposures (#2).

3.2.3 Type each exposure results on the terminal (#3).

3.2.4 Type the average of the four exposures on the terminal (#4).

3.2.5 Print the average of the four exposures (#5). The operator's manual also refers to an exposure as a burn.

3.2.6 Consult the manual for additional commands and their usage. It is recommended that at least four exposures be used in the standardization procedure.

3.2.7 If background correction is being used, concentration mode is required. Therefore, before performing any exposures, type the command "CO".

3.3 After the sixty second flush period, you may execute your command string. Each of the exposure values will appear on the terminal in concentration units (if you use the TY command in concentration mode).

3.4 After the analysis is complete, type "NS" for name standard. You will be prompted for the name of the standard. Enter the name as it appears in the ACT. This must be exact.

3.5 Aspirate the next standard and follow same procedure. Remember to use the "NS" command after each standard.

3.6 After all standards have been analyzed and named, type "SS" for save standardization. You will be prompted for element physical channel numbers. Simply push the "return" key and all standardization information for all elements in the ACT will be saved.

3.7 After executing the "SS" command, type "WA" for write the ACT. All new standardization results will now be stored in the ACT.

3.0 Standardization (continued)

3.8 Type "TS" for terminate standardization. As standardization is now complete, sample analysis can begin.

3.9 If the command file (@ ACT Name) is being used for standardization, the CRT will prompt for the specific standards.

3.9.1 After waiting 45 seconds, enter P <RETURN>.

3.9.2 After the analysis is completed, enter R2 <RETURN> to keep that analysis or PC <RETURN> to reanalyze the standard.

3.9.3 If the standard is reanalyzed, R2 <RETURN> must be entered after analysis to reenter the command file and to save the analysis.

3.9.4 This process is repeated for each standard.

4.0 Analysis

Once standardization is complete (see Section 3), verifications are performed and samples may be analyzed. A typical analysis run summary appears in Figure 1.

5.0 Instrument Shutdown

5.1 Log off the "A-to-Z" account by moving through the menus until the final choice is to indicate that you are finished with "A-to-Z". In response to the "$" prompt, type:

5.1.1 HELLO
MANAGER (for account #)
MANAGER (password)

5.1.2 From the manager account main menu, choose the system shutdown operation and follow the instructions. When the @ prompt appears after completing DUO, dismount, turn off the printer, terminal, and computer.

5.2 Press the R.F. off button and the plasma will go out. Reset all controls to the settings indicated in Section 2.3 of this procedure.

5.3 Allow argon to flow through the system for approximately five minutes. Turn off the peristaltic pump and remove the tubing from the pump winding.

5.0 Instrument Shutdown (continued)

5.4 After five minutes have elapsed, close the main valve on the argon tank. Allow torch flow and sample flow to cease before flipping toggle switches to the off position. Turn off the water recirculator.

5.5 Leave instrument main power on unless it will not be used for an extended period of time.

6.0 Quality Control (Instrumental)

6.1 Check the instrument standardization by analyzing appropriate quality control check standards as follows:

6.1.1 A quality control sample must be used daily for the initial calibration verification (ICV). A fresh dilution of this sample shall be analyzed every week thereafter to monitor their stability. If the results are not within ± 10% of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

6.1.2 Analyze the calibration blank (ICB and CCB) at a frequency of 10%. The result should be within ± contract required detection levels. If the result is not within the control level, terminate the analysis, correct the problem, and recalibrate the instrument.

6.1.3 For continuing calibration verification (CCV), analyze an appropriate instrument check standard containing the elements of interest at a frequency of 10%. This check standard is used to determine instrument drift. If agreement is not within ± 10% of the expected values, the analysis is out of control. The analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed.

6.1.4 To verify interelement and background correction factors, analyze the ICP interference check samples at the beginning and end of the sample run or a minimum of twice per eight-hour work shift, whichever is more frequent. The check sample must be analyzed initially at least five times repetitively to establish a mean value and standard deviation. Results must fall within the established control limits. If not, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the samples.

6.0 Quality Control (Instrumental) (continued)

6.1.5 To verify the calibration curve near the CRDL, a standard at a level two times the CRDL must be analyzed for certain elements.

6.1.6 Quality Control Analysis Scheme

6.1.6.1 Calibration

6.1.6.2 Initial Calibration Verification Standards (ICVA, ICVB, ...)

6.1.6.3 Initial Calibration Blank (ICB)

6.1.6.4 Interference Check Standards (ICSA, ICSAB)

6.1.6.5 2 X CRDL Standard (CRI)

6.1.6.6 Continuing Calibration Verification Standards (CCVA1, CCVB1, ...)

6.1.6.7 Continuing Calibration Blank (CCB1)

6.1.6.8 Sample Prep Blank (PB)

6.1.6.9 Laboratory Control Samples (LCSW (water), LCSS (solid))

6.1.6.10 Analyze eight samples, then run CCVA, CCVB, CCB. Thereafter, run calibration verification standards every tenth sample.

6.1.6.11 For every sample matrix, a 1/5 serial dilution must be run per 20 samples. The serial dilution must be within $\pm$ 10% of the undiluted sample for diluted sample concentration greater than 10 times the IDL.

6.1.6.12 At the conclusion of the run, the following check samples must be run as follows:

A. CCVA#, CCVB#, ...
B. CCB#
C. ICSA, ICSAB
D. CRI

Figure 1

CLP FORMAT RUN SUMMARY

I. Standardize

A. Blank (STD1)

B. STD2

C. STD3

D. STD4

II. Analysis

A. ICVA, ICVB (90 to 110% of true value)

B. ICB

C. ICSA, ICSAB (80 to 120% of true value)

D. 2 X CRDL (no requirements set to date)

E. CCVA1, CCVB1 (90 to 110% of true value)

F. CCB1

G. Prep Blank

H. LCS (80 to 120% of true value)

I. Run 8 samples (including a 1/5 serial dilution for each matrix)

J. CCVA2, CCVB2

K. CCB2

L. Run 10 samples

M. CCVA, CCVB

N. CCB

O. ICSA, ICSAB

P. 2 X CRDL

Continue pattern L - N until the end of the run and add O, P.

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE:	SOP NO: A 8705181 ~~871106R0~~
Liquid Sample Preparation for Metals - Contract Laboratory Protocol (SOW 787)	DATE INITIATED: 11/06/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 3

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Janet M Jones	Jack R. Hall	11/12/87	Mary E Tyler	11/12/87

1.0 Purpose

This Standard Operating Procedure, taken from the Contract Laboratory Protocol Statement of Work #787 (July 1987), outlines the preparation procedure for liquid samples that are to be analyzed by inductively coupled plasma (ICP), graphite furnace atomic absorption spectroscopy (GFAAS), and flame atomic absorption spectroscopy (AAS) under the Contract Laboratory Protocol. (See SOP AV871103R0 for mercury sample prep.)

2.0 Procedure

2.1 Sample Screening and Preparation Documentation

2.1.1 Chain-of-Custody: Samples are removed from the temporary storage after the appropriate checkout notebook has been signed. Group specific Chain-of-Custody forms follow the samples through the sample preparation phase. See Figure 1C.

2.1.2 Screening: Prior to preparation, the sample pH is checked and the value recorded. Additional information regarding type of preparation, date of preparation, and client identification numbers is recorded in the sample preparation logbook at this time. If the pH is found to be greater than pH 2, the samples are acidified with nitric acid and left in temporary storage for 24 hours to allow for redissolution of plated-out metals.

NOTE: A nonconformance memo is filed for each project in which the samples were received unpreserved.

2.1.3 Documentation: In addition to the project specific Chain-of-Custody form and the central preparation logbook, a project specific set of preparation worksheets is generated which is filed in the project folder. One sheet consists of test assignments generated from computer stored client

2.0 Procedure (continued)

information while the other, the sample tracking sheet, generated at the time of preparation, contains the original pH value, sample description, and observations. See Figures 1a, 1b, 1d, and 1e.

2.2 Reagents

2.2.1 Type I deionized water

2.2.2 Baker "Instra-analyzed" or equivalent acids

2.2.3 Hydrogen peroxide - reagent grade

2.3 Sample Preparation

2.3.1 Glassware preparation: Refer to SOP No. A_860619R1

2.3.2 GFAAS Sample Preparation: Shake sample and transfer 100 ml of well-mixed sample to a 250 ml beaker, add 1 ml of (1+1) HNO_3 and 2 ml 30% H_2O_2. Cover with watch glass or similar cover, heat for two hours at 95°C or until the volume is reduced to between 25 and 50 ml (make certain samples do not boil). Cool sample and filter (see Note 1) to remove insoluble material and bring back to 100 ml with deionized, distilled water. The sample is now ready for analysis.

Concentrations so determined shall be reported as "total".

2.3.3 ICP and AAS Sample Preparation: Shake sample and transfer 100 ml of a well-mixed sample to a beaker. Add 2 ml of (1+1) HNO_3 and 10 ml of (1+1) HCl to the sample. Cover with watch glass or similar cover and heat on a steam bath or hot plate until the volume has been reduced to between 25 and 50 ml (up to 2 hours maximum) making certain the sample does not boil. After this treatment, cool sample and filter to remove insoluble material that could clog the nebulizer. (See Note 1.) Adjust the volume to 100 ml with deionized, distilled water. The sample is now ready for analysis.

Concentrations so determined shall be reported as "total".

Note 1: In place of filtering, the sample after dilution and mixing may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

2.0 Procedure (continued)

2.4 Following preparation, the original samples are returned to temporary storage and the sample extracts placed in storage for the metals group. Chain-of-Custody forms are released to the metals analytical section at this time.

3.0 Quality Control

3.1 Laboratory Control Sample: Prepared with samples at a frequency of one per twenty samples, this is a standard reference material which, obtained from an independent source, is used to monitor effectiveness of sample preparation. Current sources are the NBS, the EPA, and the ERA.

3.2 Preparation Blanks: Prep blanks are prepared with every batch of samples prepared or with every twenty samples, whichever is more frequent.

3.3 Preparation Duplicates: Preparation duplicates are prepared at a minimum frequency of one per twenty samples per project.

3.4 Predigest Spikes: Predigest spikes are prepared at a minimum frequency of one per twenty samples per project. See Figure 2 for spiking information.

3.5 A QC sample initiation form is used to list samples by number and project code. When the 20th sample is reached, another form is started with QC prepped on the 1st sample on the sheet. See Figure 3.

3.6 Any sample/preparation nonconformances are noted on a nonconformance memo and distributed to the group supervisor, QC Coordinator, Lab Manager, and project file. See Figure 1e.

FIGURE 1a

SAMPLE TRACKING
METALS

MS/MSD Spike Std. Conc. _____
Volume Added _____
Spike Std. Date _____
Sample No. Spiked _____

Project Code _____
Date Prepped _____ By _____
Commercial _____ CLP _____
Type of Prep _____

Sample ID No.	Vol/Wgt AA	GF	Hg	pH	Preserve Y or N	% Solids	Matrix/ Description	Observations

ITAS-K-A_013RI

FIGURE 1b

IT ANALYTICAL SERVICES - KNOXVILLE

SAMPLE PREPARATION LOGBOOK - METALS

Sample No.	Prep Type	Weight	Prep Date of LCS and Inclusive Sample Range	Comments

FIGURE 1c

Project Code _____
EPA Case # _____
Sample No. Range _____

INTERNAL CHAIN-OF-CUSTODY FORM - METALS
IT Analytical Services-Knoxville

Sample No.	Date	Extract* Type or Original Sample?	Moved From	Moved To	Reason	Signature

* O - Original Sample
WH - Water, Hydrochloric Acid Finish
WN - Water, Nitric Acid Finish
DH - Dirt (Soil, Sediment, etc.), Hydrochloric Acid Finish
DN - Dirt (Soil, Sediment, etc.), Nitric Acid Finish

FIGURE 1d

PROJECT PREP WORKSHEET FOR GROUP LEADERS

METALS

PROJECT CODE=EVR24282 DUE DATE=05/18/87 DATE ISSUED=05/17/87 10:54

SAMPLE(S)	TY	R?	DUE DATE	PREP	ID	ANALYST	DATE
DD1293\DD1297	01	E		PA11	Hg Preservation	_____	__/__/__
	01	E		PA12	CLP-Furn.-H2O	_____	__/__/__
	01	E		PA14	CLP-I/F/G-H2O	_____	__/__/__
DD1308	31	E		PA04 701	Hg in Sediment	_____	__/__/__
	31	E		PA13	CLP-Furn.-Soil	_____	__/__/__
	31	E		PA15	CLP-I/F/G-Soil	_____	__/__/__
DD1309/DD1311	31	E		PA04 701	Hg in Sediment	_____	__/__/__
	31	E		PA13	CLP-Furn.-Soil	_____	__/__/__
	31	E		PA15	CLP-I/F/G-Soil	_____	__/__/__
DD1312	11	E		PA04 701	Hg in Sediment	_____	__/__/__
	11	E		PA13	CLP-Furn.-Soil	_____	__/__/__
	11	E		PA15	CLP-I/F/G-Soil	_____	__/__/__
DD1313/DD1318	11	E		PA04 701	Hg in Sediment	_____	__/__/__
	11	E		PA13	CLP-Furn.-Soil	_____	__/__/__
	11	E		PA15	CLP-I/F/G-Soil	_____	__/__/__

INSTRUCTIONS: USE CLP PROTOCOL
DD1293-97: LIQUID
DD1296 - SPLIT OF '93 DD1297 - SPIKE OF '93
THERE MAY BE OTHER QC - LET ME KNOW AFTER CHECKING BOTTLES

SPECIAL QC : DD1308-11: SOLID SAMPLES
DD1312-18: OIL SAMPLES - PREP AS SOLIDS

PREP-NOTES :

PREPPED BY: _____, __/__/__ APPROVED BY: _____, __/__/__

FIGURE 1e

NONCONFORMANCE MEMO
ITAS-KNOXVILLE

AA/ICP DATA REVIEW

DATE _____
PROJECT CODE _____
FILED BY _____

SAMPLE NO.(s) _____

NONCONFORMANCE: (Check applicable item(s)):

_____ (1) Method development or modification to include procedures not currently used on a regular basis (requires QA approval). (SPECIFY) _____

_____ (2) Calibration failure: (SPECIFY) _____

_____ (3) Sample identification/dilution error: (SPECIFY) _____

_____ (4) Calculation/transcription error: (SPECIFY) _____
(a) Error discovered before report to client.
(b) Error discovered after report to client.

_____ (5) Matrix spike/duplicate: _____
(a) Not recoverable due to high concentration in original sample.
(b) Not determinable due to possible sample inhomogeneity.
(c) Not determinable due to matrix effects.
(d) % Recovery / % RPD outside prescribed limits.
(e) Other: (SPECIFY) _____

_____ (6) Specified detection limit unobtainable due to: _____
(a) Matrix interferences.
(b) Limited sample volume.
(c) Blank criteria not met.
(d) Other: (SPECIFY) _____

_____ (7) Standard operating procedure not adhered to. (SPECIFY) _____

_____ (8) Holding time exceeded by _____ (days).

_____ (9) Sample received unpreserved.

_____ (10) Other: (SPECIFY) _____

CORRECTIVE ACTION TAKEN (Check applicable item(s)):

_____ (1) Error corrected by analyst. (SPECIFY) _____

_____ (2) Error corrected/resolved by QC Coordinator. (SPECIFY) _____

_____ (3) Situation noted on sample tracking sheet and appropriate lab personnel notified. (SPECIFY) _____

_____ (4) Sample processed "as is".

_____ (5) Sample preserved with _____ and let sit _____ prior to processing.

_____ (6) Samples put "on hold" until further notice.

_____ (7) Spike/standard concentration verified. New solution made if necessary.

_____ (8) Samples reanalyzed.

_____ (9) Samples reprepped and reanalyzed.

_____ (10) Client informed verbally.

_____ (11) Client informed by memo/letter.

_____ (12) Other (SPECIFY): _____

ROUTING

Title	Initials	Date	Check if Corrected
Analyst	_____	_____	_____
Group Supervisor	_____	_____	_____
QC Coordinator (if necessary)	_____	_____	_____
Assistant Lab Manager (if necessary)	_____	_____	_____

FIGURE 2

CLP SPIKES - SOW 787

ELEMENT	REQ CONC PPM	ML STD NEEDED	STOCK CONC PPM	SPIKE CONC PPM
SOLUTION #1 AA/ICP CLP SOW 787				
Aluminum	2	20	1,000	200
Arsenic	2	20	1,000	200
Barium	2	20	1,000	200
Selenium	2	20	1,000	200
Thallium	2	20	1,000	200
final volume = 100ml				
SOLUTION #2 AA/ICP CLP SOW 787				
Iron	1	10	1,000	100
Antimony	0.5	5	1,000	50
Cobalt	0.5	5	1,000	50
Lead	0.5	5	1,000	50
Manganese	0.5	5	1,000	50
Nickel	0.5	5	1,000	50
Vanadium	0.5	5	1,000	50
Zinc	0.5	5	1,000	50
Copper	0.25	2.5	1,000	25
Chromium	0.2	2	1,000	20
Beryllium	0.05	0.5	1,000	5
Cadmium	0.05	0.5	1,000	5
Silver	0.05	0.5	1,000	5
final volume = 51 ml of standards brought up to 100 ml				
SOLUTION #3 GFAAS CLP SOW 787				
Antimony	0.1	10	1,000	100
Thallium	0.05	5	1,000	50
Arsenic	0.04	4	1,000	40
Lead	0.02	2	1,000	20
Selenium	0.01	1	1,000	10
Cadmium	0.005	0.5	1,000	5
final volume = 22.5 ml of standards brought up to 100 ml				

FOR AA/ICP PREPS:

a. WATER (100 ml final volume) use 1 ml of SOLUTION #1 & 1 ml of SOLUTION #2
b. SOIL (200 ml final volume) use 2 ml of SOLUTION #1 & 2 ml of SOLUTION #2

FOR GFAAS PREPS:

a. WATER (100 ml final volume) use 0.1 ml of SOLUTION #3
b. SOIL (200 ml final volume) use 0.2 ml of SOLUTION #3

MERCURY SPIKES: 0.001 ppm is required

a. Make up a 1ppm Hg standard at the time of analysis by taking 0.05 ml of the 1,000 ppm stock standard and diluting up to 50 ml.
b. For water sample analysis: use 0.02 ml of the 1 ppm standard you made in a. (for 20 ml sample volume)
c. For soil samples: use 0.2 ml of the 1 ppm standard you made in a. (for 200 ml final volume). If you are using 250ml volumetrics for the soil prep: use 0.25 ml of the 1 ppm standard.

FIGURE 3

IT ANALYTICAL SERVICES

QC Sample Initiation Form

AA/ICP

QA/QC Sample ID: _____

Prep Code: _____ QC Type:(2) _____ Date Initiated: _____

Prep Name: _____ Date Completed: _____

Matrix: _____ Sample

Project Code:(1) _____ (Lab) ID: _____ Approved By: _____

Comments:

Prep Date/Analyst		Project Code	Sample ID	Prep/Blk
	1)			
	2)			
	3)			
	4)			
	5)			
	6)			
	7)			
	8)			
	9)			
	10)			
	11)			
	12)			
	13)			
	14)			
	15)			
	16)			
	17)			
	18)			
	19)			
	20)			
	21)			
	22)			

1) In the sample ID column, mark the original sample with an OS.

2) QC Type Designations

B = Blank R = Reference Material or Standard

D = Duplicate K = Known (stable) Standard

S = Spike

ITAS-K-A_010RO

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE:	SOP NO: A 870519R0 DATE INITIATED: 05/18/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 4

Preparation of Solid Samples for Metals - Contract Laboratory Protocol

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Katherine Whaley	Allyce K. Moore	5/20/87	Janet M. Jones	5-20-87

1.0 Purpose

Taken from the Contract Laboratory Protocol Statement of Work #785 (July 1985), this procedure describes the preparation of solid samples for analysis by inductively coupled plasma (ICP), graphite furnace atomic absorption spectroscopy (GFAAS), and flame atomic absorption spectroscopy (AAS).

2.0 Procedure

2.1 Screening and Documentation

2.1.1 Chain-of-Custody: Samples are removed from temporary storage after the appropriate checkout notebook has been signed. Project specific Chain-of-Custody forms follow the samples through the preparation phase.

2.1.2 Screening: Prior to preparation, the sample pH is checked and the value recorded on the project specific preparation worksheet. At this time, information regarding preparation type and client identification is recorded in the central sample preparation logbook, as is the date of preparation.

2.1.3 Documentation: In addition to the Chain-of-Custody forms and the central preparation logbook, project specific preparation worksheets are generated and filed in the project file. This set of worksheets consists of one sheet of test codes and instructions generated from computer stored client information and another form containing actual preparation information such as original pH, weights, volumes, sample description, and observations. Examples of each of these forms are attached.

2.0 Procedure (continued)

2.2 Reagents

2.2.1 ASTM Type II deionized water

2.2.2 Baker "Instra-analyzed" acids or equivalent

2.2.3 Hydrogen peroxide - reagent grade

2.3 Sample Preparation

2.3.1 Glassware preparation: See SOP No. A_860619R0

2.3.2 GFAAS Preparation (except for Sb): Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 gms) a 1.0 to 1.5 gm portion of sample and transfer to a beaker.

Add 10 ml of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass. Heat the sample to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5 ml of concentrated HNO_3, replace the watch glass, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 ml while maintaining a covering of solution over the bottom of the beaker.

After the second reflux step has been completed and the sample has cooled, add 2 ml of Type II water and 3 ml of 30% hydrogen peroxide (H_2O_2). Return the beaker to the hot plate for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.

Continue to add 30% H_2O_2 in 1 ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 ml 30% H_2O_2).

If the sample is being prepared for the furnace AA analysis of Sb, the flame AA or ICP analysis of Al, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Ag, Na, Tl, V, and Zn, add 5 ml of 1:1 HCl and 10 ml of Type II water, return the covered beaker to the hot plate, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 100 ml with Type II water. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v) HNO_3. Dilute the digestate 1:1 (200 ml final volume) with the deionized water. The sample is now ready for analysis.

2.0 Procedure (continued)

2.3.3 ICP/AAS/Sb (GFAAS) sample preparation: Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 gms) a 1.0 to 1.5 gm portion of sample and transfer to a beaker.

Add 10 ml of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass. Heat the sample to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5 ml of concentrated HNO_3, replace the watch glass, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 ml while maintaining a covering of solution over the bottom of the beaker.

After the second reflux step has been completed and the sample has cooled, add 2 ml of Type II water and 3 ml of 30% hydrogen peroxide (H_2O_2). Return the beaker to the hot plate for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.

Continue to add 30% H_2O_2 in 1 ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 ml 30% H_2O_2).

If the sample is being prepared for the furnace analysis of As, Be, Cd, Cr, Co, Cu, Fe, Pb, Mn, Ni, Se, Ag, Tl, V, and Zn, continue heating the acid-peroxide digestate until the volume has been reduced to approximately 2 ml, add 10 ml of Type II water, and warm the mixture. After cooling, filter through Whatman No. 42 filter paper and dilute to 100 ml with Type II water (or centrifuge the sample). The diluted digestate solution contains approximately 2% (v/v) HNO_3. Dilute the digestate 1:1 (200 ml final volume) with deionized water. For analysis, withdraw aliquots of appropriate volume, and add any required reagent or matrix modifier. The sample is now ready for analysis.

3.0 Quality Control

3.1 Laboratory Control Sample (LCS): Prepared with the samples at a frequency of one per twenty samples, this standard reference material is used to monitor the effectiveness of sample preparation. Current sources for the LCS are the EPA, the NBS, and the ERA.

3.0 Quality Control (continued)

3.2 Method Blanks: Method blanks are prepared concurrently with each set of samples at a minimum frequency of one per twenty samples each time preparation is initiated.

3.3 Preparation Duplicates: Preparation duplicates are prepared at a minimum frequency of one per twenty samples per project.

3.4 Predigest Spikes: Predigest spikes are prepared at a minimum frequency of one per twenty samples per project.

IT ANALYTICAL SERVICES - KNOXVILLE

SAMPLE PREPARATION LOGBOOK - METALS

Sample No.	Prep Type	Weight	Prep Date of LCS and Inclusive Sample Range	Comments

IT ANALYTICAL SERVICES - KNOXVILLE

SAMPLE PREPARATION WORKSHEET - METALS

Sample No.	AA Wt/Vol	GF Wt/Vol	pH	Description	Observations

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: Sample Preparation of Solid Samples for Mercury Analysis	SOP NO: AV870519R0 DATE INITIATED: 05/19/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 2

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Katherine Whaley	Alyce K. Moore	5/20/87	Janet M Jones	5-20-87

1.0 Purpose

Taken largely from Method 245.5 CLP-M of the Contract Laboratory Protocol, this procedure describes the preparation of solid samples intended for mercury analysis via the cold vapor technique. Modifications have been made to facilitate sample throughput and increase flexibility. These will be noted in the procedure.

2.0 Apparatus

2.1 Technicon BD-4 Heating Unit (Digester Block) maintained at 95°C.

2.2 75 ml volumetric digestion tubes.

2.3 200 ml volumetric flasks.

3.0 Reagents

3.1 Sulfuric acid, concentrated: Baker "Instra-Analyzed" or equivalent.

3.2 Nitric acid, concentrated: Baker "Instra-Analyzed" or equivalent

3.3 Potassium permanganate: 4% w/v solution in ASTM Type II water. This is a modification of 245.5 CLP-M which indicates that a 5% w/v solution be used.

3.4 Potassium persulfate: 4% w/v solution in ASTM Type II deionized water. This is a modification of Method 245.5 CLP-M which indicates that a 5% w/v solution be used.

4.0 Procedure

4.1 Refer to SOP No. A_860619R0 for glassware preparation.

4.2 Weigh a representative 2.0 gram portion of wet sample and place in a 75 ml volumetric digestion tube. This is a modification of Method 245.5 CLP-M instructions for placing 0.2 grams of sample into a 300 ml BOD bottle.

4.3 Add 10 ml of Type II water to the tube followed by 5 ml of concentrated sulfuric acid and 2.5 ml of concentrated nitric acid. Heat for ten minutes in the digestion block at 95°C. This is a modification of Method 245.5 CLP-M instructions calling for a two-minute heating period using a steam bath.

4.4 Add 10 ml of Type II water. Allow solution to cool, then carefully add 15 ml of 4% $KMnO_4$ solution and 8 ml of 4% $K_2S_2O_8$ solution. Return tube to digestion block and heat for an additional 30 minutes. This is a modification of Method 245.5 CLP-M which indicates that 50 ml of Type II water should be added and the 30-minute digestion carried out on a steam bath.

4.5 After allowing the sample to cool, transfer all of sample to a 200 ml volumetric flask and bring to volume with Type II water. Extracts should be analyzed no later than 48 hours following preparation. This is a modification of Method 245.5 CLP-M instruction which continues with sample treatment and analysis preceding in the same BOD bottle.

4.6 Refer to operating procedure for calibration and analysis of samples using cold vapor technique for extract analysis procedure.

5.0 Quality Control

5.1 Method Blank: Method blanks are prepared concurrently with each sample set at a minimum frequency of one per twenty samples each time sample preparation is initiated.

5.2 Preparation Duplicates: Preparation duplicates are prepared at a minimum frequency of one per twenty samples per project.

5.3 Predigest Spikes: Predigest spikes are prepared at a minimum frequency of one per twenty samples per project.

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE:	SOP NO: AP870519R0 DATE INITIATED: 05/19/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 1
Inductively Coupled Plasma - Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Katherine Whaley	Alyce K. Moore	5/20/87	Janet M Jones	5-20-87

1.0 Purpose

The following Standard Operating Procedure is presented as it appears in the Contract Laboratory Protocol, SOW #785 (July 1985) under the Method 200.7 CLP-M.

ATTACHMENT 3

Method 200.7 CLP-M*
INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROMETRIC METHOD
FOR TRACE ELEMENT ANALYSIS OF WATER AND WASTES

1. Scope and Application

1.1 Dissolved elements are determined in filtered and acidified samples. Appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account. This is especially true when dissolved solids exceed 1500 mg/L. (See 5.)

1.2 Total elements are determined after appropriate digestion procedures are performed. Since digestion techniques increase the dissolved solids content of the samples, appropriate steps must be taken to correct for potential interference effects. (See 5.)

1.3 Table 1 lists elements along with recommended wavelengths and typical estimated instrumental detection limits using conventional pneumatic nebulization. Actual working detected limits are sample dependent and as the sample matrix varies, these concentrations may also vary. In time, other elements may be added as more information becomes available and as required.

1.4 Because of the differences between various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided. Instead, the analyst is referred to the instructions provided by the manufacturer of the particular instrument.

2. Summary of Method

2.1 The method describes a technique for the simultaneous or sequential multielement determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the line are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines on

*CLP-M Modified for the Contract Laboratory Program

THALLIUM

Method 279.2 CLP-M (Atomic Absorption, furnace technique)

Optimum Concentration Range: 5-100 ug/l
Approximate Detection Limit: 1 ug/l

Preparation of Standard Solution

1. Stock solution: Dissolve 1.303g of thallium nitrate, $TlNO_3$ (analytical reagent grade) in deionized distilled water. Add 10 mL of concentrated nitric acid and dilute to 1 liter with deionized distilled water. 1 mL = 1 mg Tl (1000 mg/L).

2. Prepare dilutions of the stock solution to be used as calibration standards at the time of analysis. These solutions are also to be used for "standard additions".

3. The calibration standards must be prepared using the same type of acid and at the same concentration as will result in the sample to be analyzed after sample preparation.

Instrument Parameters (General)

1. Drying Time and Temp: 30 sec @ 125°C.
2. Ashing Time and Temp: 30 sec @ 400°C.
3. Atomizing Time and Temp: 10 sec @ 2400°C.
4. Purge Gas Atmosphere: Argon
5. Wavelength: 276.8 nm
6. Other operating parameters should be set as specified by the particular instrument manufacturer.

Notes

1. The above concentration values and instrument conditions are for a Perkin-Elmer HGA-2100, based on the use of a 20 ul injection, continuous flow purge gas and non-pyrolytic graphite and are to be used as guidelines only. Smaller size furnace devices or those employing faster rates of atomization can be operated using lower atomization temperatures for shorter time periods than the above recommended settings.

2. The use of background correction is required.

3. Nitrogen may also be used as the purge gas.

4. For every sample analyzed, verification is necessary to determine that method of standard addition is not required (see Exhibit E).

5. If method of standard addition is required, follow the procedure given in Exhibit E.

Bibliography

1. Methods for Chemical Analysis of Water and Wates (EPA-600/4-79-020), Metals - 4, Methods 204.2 (Sb), 206.2 (As), 210.2 (Be), 213.2 (Cd), 218.2 (Cr), 239.2 (Pb), 270.2 (Se), 272.2 (Ag) and 279.2 (Tl).

samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in 5.1 (and tests for their presence as described in 5.2) should also be recognized and appropriate corrections made.

3. Definitions

3.1 Dissolved — Those elements which will pass through a 0.45 um membrane filter.

3.2 Suspended — Those elements which are retained by a 0.45 um membrane filter.

3.3 Total — The concentration determined on an unfiltered sample following vigorous digestion.

3.4 Instrumental detection limits — See Exhibit E, pages 2 - 4 of the SOW #785 for the Contract Laboratory Protocol.

3.5 Sensitivity — The slope of the analytical curve, i.e. functional relationship between emission intensity and concentration.

3.6 Instrument check standard — A multielement standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis. (See 7.6.1.)

3.7 Interference check sample — A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors. (See 7.6.2.)

3.8 Quality control sample — A solution obtained from an outside source having known concentration values to be used to verify the calibration standards. (See 7.6.3.)

3.9 Calibration standards — A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). (See 7.4.)

3.10 Linear dynamic range — The concentration range over which the analytical curve remains linear as determined in Exhibit E.

3.11 Reagent blank — A volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme. (See 7.5.2.)

3.12 Calibration blank — A volume of deionized, distilled water acidified with HNO_3 and HCl. (See 7.5.1.)

3.13 Method of standard addition — The standard addition technique involves the use of the unknown and the unknown-plus-a-known amount of standard by adding known amounts of standard to one or more aliquots of the processed sample solution.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material handling data sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified (11.7, 11.8 and 11.9) for the information of the analyst.

5. Interferences

5.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

5.1.1 Spectral interferences can be categorized as 1) overlap of a spectral line from another element; 2) unresolved overlap of molecular band spectra; 3) background contribution from continuous or recombination phenomena; and 4) background contribution from stray light from the line emission of high concentration elements. The first of these effects can be compensated by utilizing a computer correction of the raw data, requiring the monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line. In addition, users of simultaneous multi-element instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but for which there is no channel in the instrument array. Listed in Table 2 are some interference effects for the recommended wavelengths given in Table 1. The data in Table 2 are intended for use only as a rudimentary guide for the indication of potential spectral interferences. For this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed. The interference information, which was collected at the Ames Laboratory[1], is expressed as

[1]Ames Laboratory, USDOE, Iowa State University, Ames Iowa 50011

analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interferent element. The suggested use of this information is as follows: Assume that arsenic (at 193.696 nm) is to be determined in a sample containing approximately 10 mg/L of of aluminum. According to Table 2, 100 mg/L of aluminum would yield a false signal for arsenic equivalent to approximately 1.3 mg/L. Therefore, 10 mg/L of aluminum would result in a false signal for arsenic equivalent to approximately 0.13 mg/L. The reader is cautioned that other analytical systems may exhibit somewhat different levels of interference than those shown in Table 2, and that the interference effects must be evaluated for each individual system. Only those interferents listed were investigated and the blank spaces in Table 2 indicate that measurable interferences were not observed from the interferent concentrations listed in Table 3. Generally, interferences were discernible if they produced peaks or background shifts corresponding to 2-5% of the peaks generated by the analyte concentrations also listed in Table 3.

At present, information on the listed silver and potassium wavelengths are not available but it has been reported that second order energy from the magnesium 383.231 nm wavelength interferes with the listed potassium line at 766.491 nm.

5.1.2 Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/ or utilization of standard addition techniques. Another problem which can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution have been used to control this problem. Also, it has been reported that better control of the argon flow rate improves instrument performance. This is accomplished with the use of mass flow controllers.

5.1.3 Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique, however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

5.2 For each group of samples of a similar matrix type and concentration (i.e., low, medium) for each Case of samples, or for each 20 samples received, whichever is more frequent, the following tests must be performed prior to reporting concentration data for analyte elements.

5.2.1 Serial dilution — If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrument detection limit after dilution), an analysis of a 1:4 dilution must agree within 10 percent of the original determination. Serial dilution results must be reported on QC Report Form IX. Samples identified as Field Blanks cannot be used for serial dilution analysis.

If the dilution analysis is not within 10%, a chemical or physical interference effect should be suspected, and the data must be flagged with an "E".

6. Apparatus

6.1 Inductively Coupled Plasma-Atomic Emission Spectrometer.

6.1.1 Computer controlled atomic emission spectrometer with background correction.

6.1.2 Radiofrequency generator.

6.1.3 Argon gas supply, welding grade or better.

6.2 Operating conditions — Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument. All measurements must be within the instrument linear range where correction factors are valid. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain quality control data confirming instrument performance and analytical results.

7. Reagents and standards

7.1 Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. Redistilled acids are acceptable.

7.1.1 Acetic acid, conc. (sp gr 1.06).

7.1.2 Hydrochloric acid, conc. (sp gr 1.19).

7.1.3 Hydrochloric acid, (1+1): Add 500 mL conc. HCl (sp gr 1.19) to 400 mL deionized, distilled water and dilute to 1 liter.

7.1.4 Nitric acid, conc. (sp gr 1.41).

7.1.5 Nitric acid, (1+1): Add 500 mL conc. HNO_3 (sp gr 1.41) to 400 mL deionized, distilled water and dilute to 1 liter.

7.2 Deionized, distilled water: Prepare by passing distilled water through a mixed bed of cation and anion exchange resins. Use deionized, distilled water for the preparation of all reagents, calibration standards and as dilution water. The purity of this water must be equivalent to ASTM Type II reagent water of Specification D 1193 (14,6).

7.3 Standard stock solutions may be purchased or prepared from ultra high purity grade chemicals or metals. All salts must be dried for 1 h at 105° unless otherwise specified.

(CAUTION: Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.) Typical stock solution preparation procedures follow:

7.3.1 Aluminum solution, stock, 1 mL = 100 ug Al: Dissolved 0.100 g of aluminum metal in an acid mixture of 4 mL of (1+1) HCl and 1 mL of conc. HNO_3 in a beaker. Warm gently to effect solution. When solution is complete, transfer quantitatively to a liter flask, add an additional 10 mL of (1+1) HCl and dilute to 1000 mL with deionized, distilled water.

7.3.2 Antimony solution stock, 1 mL = 100 ug Sb: Dissolve 0.2669 g $K(SbO)C_4H_4O_6$ in deionized distilled water, add 10 mL (1+1) HCl and dilute to 1000 mL with deionized, distilled water.

7.3.3 Arsenic solution, stock, 1 mL = 100 ug As: Dissolve 0.1320 g of As_2O_3 in 100 mL of deionized, distilled water containing 0.4 g NaOH. Acidify the solution with 2 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.4 Barium solution, stock, 1 mL = 100 ug Ba: Dissolve 0.1516 g $BaCl_2$ (dried at 250°C for 2 hrs) in 10 mL deionized, distilled water with 1 mL (1+1) HCl. Add 10.0 mL (1+1) HCl and dilute to 1,000 mL with deionized, distilled water.

7.3.5 Beryllium solution, stock, 1 mL = 100 ug Be: Do not dry. Dissolve 1.966 g $BeSO_4 \cdot 4H_2O$, in deionized, distilled water, add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.6 Boron solution, stock, 1 mL = 100 ug B: Do not dry. Dissolve 0.5716 g anhydrous H_3BO_3 in deionized, distilled water and dilute to 1,000 mL. Use a reagent meeting ACS specifications, keep the bottle tightly stoppered and store in a desiccator to prevent the entrance of atmospheric moisture.

7.3.7 Cadmium solution, stock, 1 mL = 100 ug Cd: Dissolve 0.1142 g CdO in a minimum amount of (1+1) HNO_3. Heat to increase rate of dissolution. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.8 Calcium solution, stock, 1 mL = 100 ug Ca: Suspend 0.2498 g $CaCO_3$ dried at 180°C for 1 h before weighing in deionized, distilled water and dissolve cautiously with a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.9 Chromium solution, stock, 1 mL = 100 ug Cr: Dissolve 0.1923 g of CrO_3 in deionized, distilled water. When solution is complete acidify with 10 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.10 Cobalt solution stock, 1 mL = 10 ug Co: Dissolve 0.1000 g of cobalt metal in a minimum amount of (1+1) HNO_3. Add 10.0 mL (1+1) HCl and dilute to 1,000 mL with deionized, distilled water.

7.3.11 Copper solution, stock, 1 mL = 100 ug Cu: Dissolve 0.1252 g CuO in a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.12 Iron solution, stock, 1 mL = 100 ug Fe: Dissolve 0.1430 g Fe_2O_3 in a warm mixture of 20 mL (1+1) HCl and 2 mL of conc. HNO_3. Cool, add an additional 5 mL of conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.13 Lead solution, stock, 1 mL = 100 ug Pb: Dissolve 0.1599 g $Pb(NO_3)_2$ in a minimum amount of (1+1) HNO_3. Add 10.0 mL of conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.14 Magnesium solution, stock, 1 mL = 100 ug Mg: Dissolve 0.1658 g MgO in a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.15 Manganese solution, stock, 1 mL = 100 ug Mn: Dissolve 0.1000 g of manganese metal in the acid mixture, 10 mL conc. HCl and 1 mL conc. HNO_3, and dilute to 1,000 mL with deionized, distilled water.

7.3.16 Molybdenum solution, stock, 1 mL = 100 ug Mo: Dissolve 0.2043 g $(NH_4)_2MoO_4$ in deionized, distilled water and dilute to 1,000 mL.

7.3.17 Nickel solution, stock, 1 mL = 100 ug Ni: Dissolve 0.1000 g of nickel metal in 10 mL hot conc. HNO_3, cool and dilute to 1,000 mL with deionized, distilled water.

7.3.18 Potassium solution, stock, 1 mL = 100 ug K: Dissolve 0.1907 g KCl, dried at 110°C, in deionized, distilled water. Dilute to 1,000 mL.

7.3.19 Selenium solution, stock, 1 mL = 100 ug Se: Do not dry. Dissolve 0.1727 g H_2SeO_3 (actual assay 94.6%) in deionized, distilled water and dilute to 1,000 mL.

7.3.20 Silica solution, stock, 1 mL = 100 ug SiO_2: Do not dry. Dissolve 0.4730 g $Na_2SiO_3 \cdot 9H_2O$ in deionized, distilled water. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.21 Silver solution, stock, 1 mL = 100 ug Ag: Dissolve 0.1575 g $AgNO_3$ in 100 mL of deionized, distilled water and 10 mL conc. HNO_3. Dilute to 1,000 mL with deionized, distilled water.

7.3.22 Sodium solution, stock, 1 mL = 100 ug Na: Dissolve 0.2542 g NaCl in deionized, distilled water. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.23 Thallium solution, stock, 1 mL = 100 ug Tl: Dissolve 0.1303 g $TlNO_3$ in deionized, distilled water. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.24 Vanadium solution, stock, 1 mL = 100 ug V: Dissolve 0.2297 NH_4VO_3 in a minimum amount of conc. HNO_3. Heat to increase rate of dissolution. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.25 Zinc solution, stock, 1 mL = 100 ug Zn: Dissolve 0.1245 g ZnO in a minimum amount of dilute HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.4 Mixed calibration standard solutions — Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. (See 7.4.1 thru 7.4.5.) Add 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl and dilute to 100 mL with deionized, distilled water. (See Notes 1 and 6.) Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards that the elements are compatible and stable. Transfer the mixed standard solutions to a FEP fluorocarbon or unused polyethylene bottle for storage. Fresh mixed standards should be prepared as needed with the realization that concentration

can change on aging. Calibration standards must be initially verified using a quality control sample and monitored weekly for stability (see 7.6.3). Although not specifically required, some typical calibration standard combinations follow when using those specific wavelengths listed in Table 1.

7.4.1 Mixed standard solution I — Manganese, beryllium, cadmium, lead, and zinc.

7.4.2 Mixed standard solution II — Barium, copper, iron, vanadium, and cobalt.

7.4.3 Mixed standard solution III — Molybdenum, silica, arsenic, and selenium.

7.4.4 Mixed standard solution IV — Calcium, sodium, potassium, aluminum, chromium and nickel.

7.4.5 Mixed standard solution V — Antimony, boron, magnesium, silver, and thallium.

NOTE 1: If the addition of silver to the recommended acid combination results in an initial precipitation add 15 mL of deionized distilled water and warm the flask until the solution clears. Cool and dilute to 100 mL with deionized, distilled water. For this acid combination the silver concentration should be limited to 2 mg/L. Silver under these conditions is stable in a tap water matrix for 30 days. Higher concentrations of silver require additional HCl.

7.5 Two types of blanks are required for the analysis. The calibration blank (3.13) is used in establishing the analytical curve while the reagent blank (preparation blank, 3.12) is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing.

7.5.1 The calibration blank is prepared by diluting 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl to 100 mL with deionized, distilled water. (See Note 6.) Prepare a sufficient quantity to be used to flush the system between standards and samples.

7.5.2 The reagent blank (or preparation blank - See Exhibit E) must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

7.6 In addition the calibration standards, an instrument check standard (3.6), an interference check sample (3.7) and a quality control sample (3.8) are also required for the analyses.

7.6.1 The instrument check standard for continuing calibration verification is prepared by the analyst by combining compatible elements at a concentration equivalent to the midpoint of their respective calibration curves. (See 10.1.3.)

7.6.2 The interference check sample is prepared by the analyst, or obtained from EPA if available (Exhibit E) of the Contract Laboratory Protocol.

7.6.3 The quality control sample for the initial calibration verification should be prepared in the same acid matrix as the calibration standards and in accordance with the instructions provided by the supplier. EPA will either supply a quality control sample or information where one of equal quality can be procured. (See 10.1.1.)

8. Procedure

8.1 Set up instrument with proper operating parameters established in Section 6.2. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 30 min. of operation prior to calibration.

8.2 Initiate appropriate operating configuration of computer.

8.3 Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using mixed calibration standard solutions such as those described in Section 7.4. Flush the system with the calibration blank (7.5.1) between each standard. (See NOTE 7.) (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.)

NOTE 7: For boron concentrations greater than 500 ug/L extended flush times of 1 to 2 minutes may be required.

8.4 Begin the sample run flushing the system with the calibration blank solution (7.5.1) between each sample. (See NOTE 7.) Analyze the instrument check standard (7.6.1) and the calibration blank (7.5.1) each 10 samples.

9. Calculation

9.1 Reagent blanks (preparation blanks) should be treated as specified in Exhibit E of the SOW #785 Contract Laboratory Protocol.

9.2 If dilutions were performed, the appropriate factor must be applied to sample values.

9.3 Data must be reported in ug/L for liquid samples.

10. Quality Control (Instrumental)

10.1 Check the instrument standardization by analyzing appropriate quality control check standards as follows:

10.1.1 A quality control sample (7.6.3) must be used daily for the initial calibration verification (See Exhibit E). A fresh dilution of this sample shall be analyzed every week thereafter to monitor their stability. If the results are not within ±10% of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

10.1.2 Analyze the calibration blank (7.5.1) at a frequency of 10%. The result should be within ± contract required detection levels (Exhibit C). If the result is not within the control level, terminate the analysis, correct the problem and recalibrate the instrument (See Exhibit E).

10.1.3 For continuing calibration verification, analyze an appropriate instrument check standard (7.6.1) containing the elements of interest at a frequency of 10%. This check standard is used to determine instrument drift. If agreement is not within ±10% of the expected values, the analysis is out of control. The analysis must be terminated, the problem corrected, the instrument recalibrated, and the preceding 10 samples reanalyzed (See Exhibit E).

10.1.4 To verify interelement and background correction factors analyze the ICP interference check sample (7.6.2) at the beginning, and end of the sample run or a minimum of twice per 8 hour work shift whichever is more frequent. The check sample must be analyzed initially at least 5 times repectively to establish a mean value and standard deviation. Results must fall within the established control limits. If not, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the samples (See Exhibit E).

11. Bibliography

1. Winge, R. K., V.J. Peterson, and V.A. Fassel, "Inductively Coupled Plasma-Atomic Emission Spectroscopy Prominent Lines," EPA-600/4-79-017.

2. Winefordner, J.D., "Trace Analysis: Spectroscopic Methods for Elements," Chemical Analysis, Vol. 46, pp. 41-42.

3. Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA-600/4-79-019.

4. Garbarino, J.R. and Taylor, H.E., "An Inductively-Coupled Plasma Atomic Emission Spectrometric Method for Routine Water Quality Testing," Applied Spectroscopy 33, No. 3(1979).

5. "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020.

6. Annual Book of ASTM Standards, Part 31.

7. "Carcinogens - Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, Aug. 1977.

8. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206, (Revised, January 1976).

9. "Safety in Academic Chemistry Laboratories, American Chemical Society Publications, Committee on Chemical Safety, 3rd Edition, 1979.

10. "Inductively Coupled Plasma-Atomic Emission Spectrometric Method of Trace Elements Analysis of Water and Waste", Method 200.7 modified by CLP Inorganic Data/Protocol Review Committee; original method by Theodore D. Martin, EMSL/Cincinnati.

TABLE 1 - RECOMMENDED WAVELENGTHS(2) AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Element	Wavelength, nm(1)	Estimated Detection Limit, ug/L(2)
Aluminum	308.215	45
Antimony	206.833	32
Arsenic	193.696	53
Barium	455.403	2
Beryllium	313.042	0.3
Boron	249.773	5
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Magnesium	279.079	30
Manganese	257.610	2
Molybdenum	202.030	8
Nickel	231.604	15
Potassium	766.491	see(3)
Selenium	196.026	75
Silica (SiO_2)	288.158	58
Silver	328.068	7
Sodium	588.995	29
Thallium	190.864	40
Vanadium	292.402	8
Zinc	213.856	2

(1) The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelength may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. (See 5.1.1). The use of alternate wavelengths must be reported (in nm) with the sample data.

(2) The estimated instrumental detection limits as shown are taken from "Inductively Coupled Plasma-Atomic Emission Spectroscopy-Prominent Lines," EPA-600/4-79-017. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

(3) Highly dependent on operating conditions and plasma position.

TABLE 2. EXAMPLE OF ANALYTE CONCENTRATION EQUIVALENTS (mg/L) ARISING FROM INTERFERENTS AT THE 100 mg/L LEVEL

Analyte	Wavelength, nm	Interferent									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Boron	249.773	0.04	--	--	--	0.32	--	--	--	--	--
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Silicon	288.158	--	--	0.07	--	--	--	--	--	--	0.01
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

TABLE 3. INTERFERENT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR INTERFERENCE MEASUREMENTS IN TABLE 2 (EXHIBIT D)

Analytes	(mg/L)	Interferents	(mg/L)
Al	10	Al	1000
As	10	Ca	1000
B	10	Cr	200
Ba	1	Cu	200
Be	1	Fe	1000
Ca	1	Mg	1000
Cd	10	Mn	200
Co	1	Ni	200
Cr	1	Ti	200
Cu	1	V	200
Fe	1		
Mg	1		
Mn	1		
Mo	10		
Na	10		
Ni	10		
Pb	10		
Sb	10		
Se	10		
Si	1		
Tl	10		
V	1		
Zn	10		

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: Hazardous Substance List (HSL) Analysis - Metals Contract Laboratory Protocol	SOP NO: A 870520R0 DATE INITIATED: 05/18/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 11

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Katherine Whaley	Alyce K. Moore	5/20/87	Janet M Jones	5-20-87

1.0 Purpose

Taken from the Contract Laboratory Protocol Statement of Work (SOW) #785 (July 1985) this Standard Operating Procedure addresses the handling of HSL analysis requests for metals from sample preparation through sample analysis and data package presentation. Data package forms and parameters presented herein reflect current useage and may change in both content and number with subsequent SOW revisions. This procedure will address the following items: 1) Current data package forms; 2) General sample preparation scheme; 3) Current HSL metals list and methods; 4) Data package contents; 5) Provisions for problems; and, 6) Quality assurance and quality control (QA/QC) requirements for SOW 785.

2.0 HSL Metals List and Methods of Analysis

The methods appear in order of priority for useage with those in parentheses representing method numbers from the September 1986 edition of SW-846. Footnotes appear when additional information is required. The following qualifiers appear to further identify methods: (ICP) Inductively Coupled Plasma, (AA) Direct Aspiration - Flame, (GFAAS) Graphite Furnace

3.0 Overview of Sample Preparation

Table II presents the summarized sample preparation scheme. Refer to the individual operating procedures for more detailed descriptions of preparation.

4.0 Sample Data Package Contents

4.1 A completed data package will include the following elements:

- Case narrative
- Cover page - inorganic analysis data package
- Sample results on Form I
- Completed contractual QC Forms II through XIII
- Copies of ICP, GFAAS, Hg digestion logs or comparable worksheets
- Analytical raw data
- Copies of traffic reports and Chain-of-Custody forms

4.2 Blank forms are attached.

4.3 Comments

4.3.1 Cover page: The cover page for the inorganic analysis data package includes general comments, Statement of Work (SOW) number, unique QC report number, sample client cross reference numbers in alpha-numeric order, footnotes used in the data package, and the statement on use of ICP background and inter-element corrections for the samples. The SOW number defines the Statement of Work used to obtain the reported values. The QC report number is a unique number assigned by the contractor to all Quality Control Data Reports generated in conjunction with and supportive of a particular set of sample analyses. It is intended that the presence of the QC report number on the cover page and on Form I data sheets will establish linkage and traceability of the sample analytical data to the associated quality control data.

4.3.2 Forms XI through XIII are generated quarterly for instrument parameter verification.

4.3.3 Analytical raw data includes all information needed to reconstruct sample life from preparation to report.

5.0 Potential Problems and Provisions for Dealing with Them

5.1 Instrument malfunction: If the ICP unit malfunctions, those elements affected will be analyzed for by AAS.

5.2 Table III presents a list of potential problems and how they will be dealt with. Attempts will be made to provide flexibility in all areas.

5.3 Solid samples will not be mixed and pulverized. Reasonable attempts will be made to obtain a homogeneous aliquot without destroying sample integrity.

5.4 Problems will be documented in the case narrative and/or nonconformance memos.

6.0 QA/QC Requirements

The following outline lists the topics to be covered in this section.

6.1 Quarterly Verification of Instrument Parameters
6.2 Initial Calibration and Calibration Verification
6.3 Continuing Calibration Verification
6.4 Preparation Blank Analysis
6.5 Interference Check Sample Analysis
6.6 ICP Serial Dilution Analysis
6.7 Matrix Spike Analysis
6.8 Duplicate Sample Analysis
6.9 Furnace AA QC Analysis
6.10 Laboratory Control Sample Analysis

6.1 Quarterly Verification of Instrument Parameters

6.1.1 Instrument Detection Limit (IDL) Determination

6.1.1.1 IDL's must be determined prior to the analysis of any field samples under the contract and at least quarterly for each instrument.

6.1.1.2 IDL's must meet the Contract Required Detection Limits (CRDL) specified in Table IV.

6.1.1.3 IDL's are three times the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration 3-5 times IDL, with seven consecutive measurements per day.

6.0 QA/QC Requirements (continued)

6.1.1.4 QC Report Form XI and the documentation for IDL determinations must be submitted as part of the data package.

6.1.1.5 For each case, IDL's must be reported on QC Report Form VII.

6.1.1.6 If multiple instruments of the same type are used for the analysis of an element within a case, the highest IDL for that instrument type must be reported on the QC Report Form VII for that case.

6.1.2 Linear Range Analysis

6.1.2.1 Linear range verification check standard must be analyzed and reported quarterly for each element on QC Form XII.

6.1.2.2 Analytically determined concentration of this standard must be written ± 5% of the true value.

6.1.2.3 The concentration of the standard run defines the upper limit of the ICP linear range beyond which results cannot be reported without dilution.

6.1.2.4 When an analyte concentration exceeds the linear range, reanalysis of the prepared sample, after appropriate dilution, is required.

6.1.3 Interelement Correction Factors

6.1.3.1 Determine as per instrument manufacturer's instructions.

6.1.3.2 Report correction factors on QC Report Form XII.

6.2 Initial Calibration and Calibration Verification

6.2.1 Calibration

6.2.1.1 Instruments must be calibrated each time the instrument is set up.

6.0 QA/QC Requirements (continued)

6.2.1.2 AA Systems

- Blank + 3 calibration standards
- One standard must be at the CRDL (except for Hg)

6.2.1.3 ICP Systems

- Follow instrument manufacturer's recommended procedures (minimum: blank + 1 standard)
- To verify linearity near the CRDL, a 2X CRDL standard must be analyzed at the beginning and end of each sample analysis run, or a minimum of twice per 8 hour working shift, whichever is more frequent (for all ICP elements except Al, Ba, Ca, Fe, Mg, Na, and K).

6.2.2 Calibration Verification

6.2.2.1 The accuracy of the initial instrument calibration must be verified and documented for every analyte by the analysis of Initial Calibration Verification Solutions (ICVS).

6.2.2.2 If an ICVS is not available from EPA or where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for calibration, but within the calibration range.

6.2.2.3 Independent standard: Standard composed of analytes from a different source than those used in the standards for the initial instrument calibration.

6.2.2.4 The ICVS must be run at each wavelength used for analysis.

6.2.2.5 The ICVS must fall within the specified control limits (Table V).

6.2.2.6 ICVS results must be recorded on QC Form II.

6.0 QA/QC Requirements (continued)

6.2.3 Calibration Blank

6.2.3.1 Must be analyzed each time instrument calibrated

6.2.3.2 Must be analyzed at the beginning and the end of the run, and at a frequency of 10% during the run.

6.2.3.3 Results must be recorded on QC Form III.

6.2.3.4 Blank results are to be reported down to the IDL.

6.2.3.5 If result is greater than CRDL, terminate analysis, correct the problem, and recalibrate.

6.3 Continuing Calibration Verification (CCV)

6.3.1 CCV must be performed for each analyte at a frequency of 10% or every two hours during an analysis run, whichever is more frequent.

6.3.2 CCV must also be analyzed for each analyte at the beginning and end of the analysis run.

6.3.3 The same continuing calibration standard must be used throughout the analysis run for a particular case.

6.3.4 One of the following standards must be used for continuing calibration verification:

1. EPA solution
2. NBS SRM 1643a
3. Contractor prepared solution

6.3.5 If CCV results exceed the specified control limits (Table V), the instrument must be recalibrated and the preceding 10 samples reanalyzed for the analytes affected.

6.3.6 CCV results must be recorded on Form II.

6.4 Preparation Blank Analysis

6.4.1 Preparation Blank (PB) - deionized, distilled H_2O processed through every step of a sample preparation procedure.

6.0 QA/QC Requirements (continued)

6.4.2 For every 20 samples received or with each batch of samples digested, whichever is more frequent, at least one Preparation Blank must be prepared and analyzed for each procedure performed in the analysis of a case of samples.

6.4.3 Batch: A group of samples prepared at the same time.

6.4.4 Results are to be reported in µg/L on QC Form III.

6.4.5 The data package must contain the results of all the Preparation Blank analyses associated with the samples in that case.

6.4.6 If the concentration of the blank is > CRDL, all associated samples which are < 10x the blank concentration must be redigested and reanalyzed (exception: AQ-SOL field blank).

6.4.7 Sample values are not to be corrected for the blank value.

6.5 ICP Interference Check Sample Analysis

Frequency: Beginning and end of each sample analysis run (minimum 2x/8 hours)

6.5.1 ICP Interference Check Samples (ICS) supplied by EPA (EMSL-LV).

6.5.2 ICS results must fall within the control limit of ± 20% of the EPA supplied true value for the analytes included in the ICS. Otherwise, terminate the analysis, correct the problem, recalibrate, reverify the calibration, and reanalyze the samples.

6.5.3 If EPA ICS is not available, an independent ICS must be prepared with the interferent and analyte concentrations at the levels specified in Table VII.

6.5.4 For the independent standard, the mean value and standard deviation must be established by initially analyzing the ICS at least 5x repetitively for each parameter listed on Form IV.

6.5.5 Results of the contractor prepared ICS must fall within the control limit of ± 20% of the established mean value.

6.5.6 ICS result must be recorded on Form IV.

6.0 QA/QC Requirements (continued)

6.6 ICP Serial Dilution Analysis

6.6.1 Must be performed on each group of samples of a similar matrix type (i.e., water, soil) for each case of samples or for each 20 samples received, whichever is more frequent.

6.6.2 Samples identified as field blanks cannot be used for serial dilution analysis.

6.6.3 An analysis of a 1:4 dilution must agree within 10% of the original determination on the undiluted sample when the analyte concentration is minimally a factor of 10x IDL after dilution.

6.6.4 If the dilution analysis is not within 10%, the data must be flagged with an "E".

6.6.5 Serial dilution results must be reported on QC Report Form IX.

6.7 Spiked Sample Analysis

6.7.1 Predigestion/predistillation spike

6.7.2 At least one spiked sample analysis must be performed on each group of samples of a similar matrix type for each case of samples or for each 20 samples received, whichever is more frequent.

6.7.3 Samples identified as field blanks cannot be used for spiked sample analysis.

6.7.4 Analyte spike levels are specified in Table VI.

6.7.5 If spike recovery is not written within the limits of 75- 125%, all data associated with that spike must be flagged "N" (exception: when sample concentration is 4x spike concentration).

6.7.6 $$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

where: SSR = spiked sample result
SR = sample result (where SR < IDL, use SR = 0)
SA = spike added

6.0 QA/QC Requirements (continued)

6.7.7 Spiked sample results must be reported on Form V.

6.7.8 If two analytical methods are used to obtain the reported values for the same element for a case of samples, spike samples must be run by each method used.

6.8 Duplicate Sample Analysis

6.8.1 At least one duplicate sample must be analyzed from each group of samples of a similar matrix type for each case of samples or for each 20 samples received, whichever is more frequent.

6.8.2 Samples identified as field blanks cannot be used for duplicate sample analysis.

6.8.3 If two analytical methods (i.e., ICP, AA) are used to obtain the reported values for the same element for a case of samples, duplicate samples must be run by each method used.

6.8.4 $$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where: RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (duplicate)

6.8.5 Duplicate sample results must be reported on Form VI.

6.8.6 Control limits: $\pm$ 20% RPD for sample results > 5x CRDL
$\pm$ CRDL for sample results < 5x CRDL
$\pm$ CRDL for one result > 5x CRDL, the other < 5x CRDL
if either result < CRDL, RPD is "N.C."

6.8.7 Flag all associated results for RPD's which exceed the control limits with an "*" on Form I.

6.9 Furnace Atomic Absorption QC Analysis

6.9.1 Duplicate Injections

6.9.1.1 Required for all furnace analyses except during full MSA.

6.0 QA/QC Requirements (continued)

6.9.1.2 Raw data must contain both readings, the average value and the RSD or CV average result must be reported on Form I.

6.9.1.3 For concentrations > CRDL, duplicate injection readings must agree within 20% RSD or CV or the sample must be rerun once.

6.9.1.4 If after the third injection the readings are still out, flag the value with a "M" on Form I.

6.9.2 Analytical Spikes (Post-Digest)

6.9.2.1 All furnace analyses for each sample requires at least a single analytical spike.

6.9.2.2 Analytical spikes are not required on predigest spike sample.

6.9.2.3 Percentage recovery determines how the sample will be quantitated (refer to Figure 1).

6.9.3 Multiple Standard Additions (MSA) Requirements

6.9.3.1 Data must be within linear range as determined by the calibration curve.

6.9.3.2 The original sample and the three spikes must be analyzed consecutively.

6.9.3.3 Only single injections are required.

6.9.3.4 Spikes should be prepared such that:

Spike 1 is ≈ 50% of the sample absorbance
Spike 2 is ≈ 100% of the sample absorbance
Spike 3 is ≈ 150% of the sample absorbance

6.9.3.5 Raw data must include slope, intercept and correlation coefficient (r).

6.9.3.6 MSA results must be reported on Form VIII.

6.0 QA/QC Requirements (continued)

6.9.3.7 Results obtained by MSA must be flagged "s" on Form I.

6.9.3.8 If r < 0.995, the MSA must be repeated once. If 2nd r is still < 0.995, then flag Form I result with "+".

6.9.3.9 See Figure I for flow chart of furance analysis scheme.

6.10 Laboratory Control Sample (LCS) Analysis

The LCS must be analyzed for each analyte using the same methods employed for samples (preparation and analysis).

6.10.1 Aqueous (AQ) LCS

6.10.1.1 One AQ LCS must be prepared and analyzed for every 20 samples received, or for each batch of samples digested, whichever is more frequent.

6.10.1.2 For Hg, AQ LCS is not required.

6.10.1.3 Results must be reported on QC Form VII.

6.10.1.4 If results (%R) exceed control limits of 80-120%, analyses must be terminated, the problem corrected, and the samples associated with that LCS reanalyzed.

6.10.2 Solid Sample Matrix

Laboratories participating in the CLP program (have a government contract) receive a solid material to be prepared on a monthly basis. As of the writing of SOW 785, no control limits had been set. Currently, this laboratory is using a liquid concentrate standard reference material with certified values to verify sample preparation. It is prepared with samples at a frequency of one per twenty samples per project. This may change depending on SOW revisions and availability of solid material with control limits.

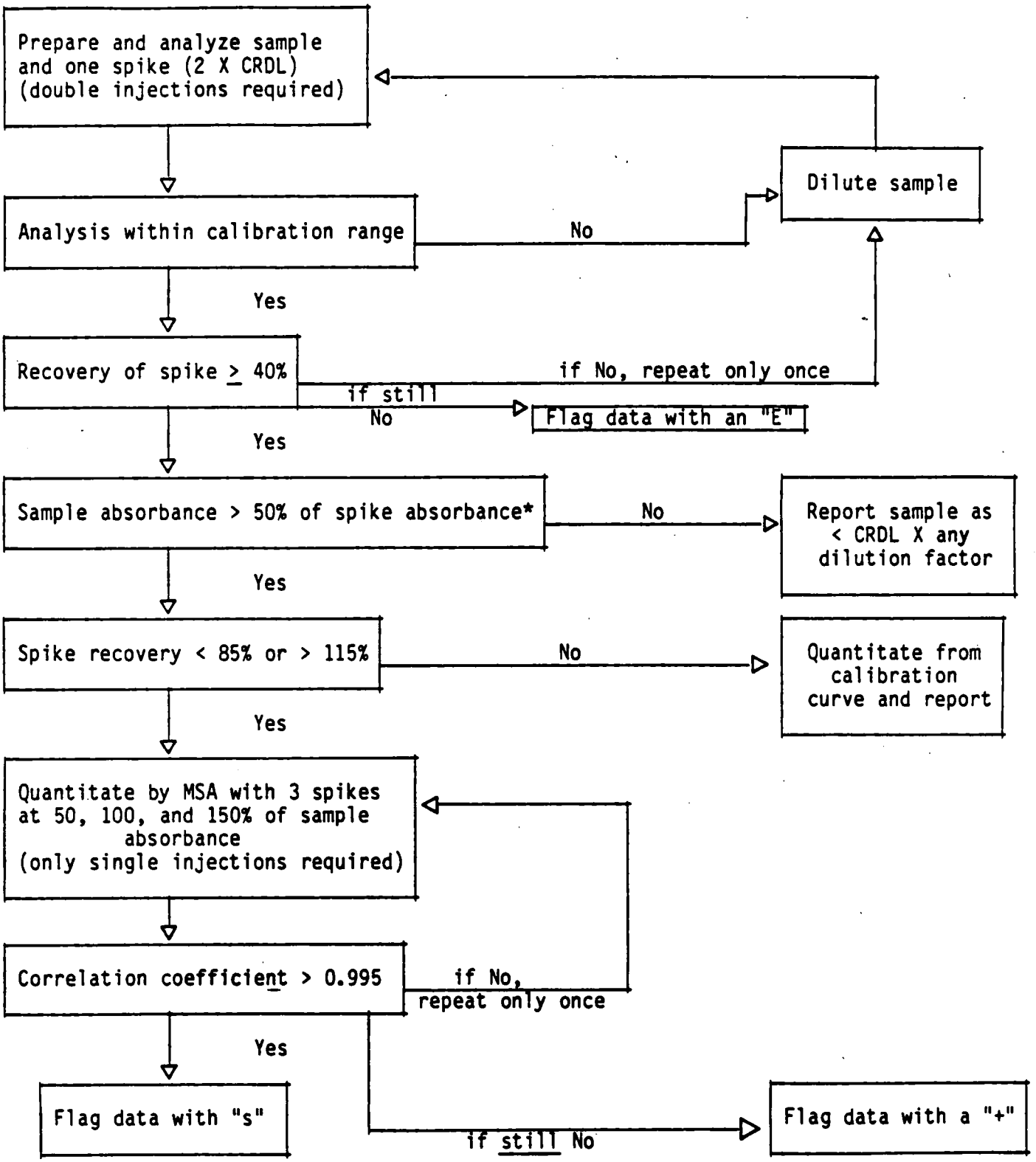
TABLE VII

INTERFERENT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR ICP INTERFERENCE CHECK SAMPLE

Analytes	(mg/L)	Interferents	(mg/L)
Ba	0.5	Al	500
Be	0.5	Ca	500
Cd	1.0	Fe	200
Co	0.5	Mg	500
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	1.0		
V	0.5		
Zn	1.0		

FIGURE 1

FURNACE ATOMIC ABSORPTION ANALYSIS SCHEME



*Spike absorbance defined as (absorbance of spike sample) minus (absorbance of the sample)

U.S. EPA Contract Laboratory Program
Sample Management Office
P.O. Box 818 - Alexandria, VA 22313
703/557-2490 FTS: 8-557-2490

Date _____

COVER PAGE
INORGANIC ANALYSES DATA PACKAGE

Lab Name ITAS-Knoxville Case No. _____

SOW No. 785 Q.C. Report No. _____

Sample Numbers

EPA No.	Lab ID No.	EPA No.	Lab ID No.

Comments: AV: Method symbol for mercury analysis via cold vapor technique.

ICP interelement and background corrections applied? Yes ____ No ____.

If yes, corrections applied before _____ or after _____ generation of raw data.

Footnotes:

NR - Not required by contract at this time

Form I:

Value - If the result is a value greater than or equal to the instrument detecion limit but less than the contract-required detection limit, report the value in brackets (i.e., [10]). Indicate the analytical method used with P (for ICP), A (for Flame AA) or F (for Furnace AA).

U - Indicates element was analyzed for but not detected. Report with the instrument detection limit value (e.g., 10U).

E - Indicates a value estimated or not reported due to the presence of interference. Explanatory note included on cover page.

S - Indicates value determined by Method of Standard Addition.

N - Indicates spike sample recovery is not within control limits.

* - Indicates duplicate analysis is not within control limits.

+ - Indicates the correlation coefficient for method of standard addition is less than 0.995

M - Indicates duplicate injection results exceeded control limits.

Indicate method used: P for ICP; A for Flame AA and F for Furnace.

Form I

U.S. EPA Contract Laboratory Program
Sample Management Office
P.O. Box 818 - Alexandria, VA 22313
703/557-2490 FTS: 8-557-2490

EPA Sample No.

Date _____

INORGANIC ANALYSIS DATA SHEET

LAB NAME _____ CASE NO. _____

SOW NO. _____

LAB SAMPLE ID. NO. _____ QC REPORT NO. _____

Elements Identified and Measured

Concentration: Low _____ Medium _____

Matrix: Water _____ Soil _____ Sludge _____ Other _____

ug/L or mg/kg dry weight (Circle One)

1.	Aluminum	13.	Magnesium
2.	Antimony	14.	Manganese
3.	Arsenic	15.	Mercury
4.	Barium	16.	Nickel
5.	Beryllium	17.	Potassium
6.	Cadmium	18.	Selenium
7.	Calcium	19.	Silver
8.	Chromium	20.	Sodium
9.	Cobalt	21.	Thallium
10.	Copper	22.	Vanadium
11.	Iron	23.	Zinc
12.	Lead	Precent Solids (%)	

Cyanide _____

Footnotes: For reporting results to EPA, standard result qualifiers are used as defined on Cover Page. Additional flags or footnotes explaining results are encouraged. Definition of such flags must be explicit and contained on Cover Page, however.

Comments: _____

Lab Manager _____

Form II

Q. C. Report No. _____

INITIAL AND CONTINUING CALIBRATION VERIFICATION[3]

LAB NAME _____ CASE NO. _____

SOW NO. _____

DATE _____ UNITS _____

Compound	Initial Calib.[1]			Continuing Calibration[2]					
Metals:	True Value	Found	%R	True Value	Found	%R	Found	%R	Method[4]
1. Aluminum									
2. Antimony									
3. Arsenic									
4. Barium									
5. Beryllium									
6. Cadmium									
7. Calcium									
8. Chromium									
9. Cobalt									
10. Copper									
11. Iron									
12. Lead									
13. Magnesium									
14. Manganese									
15. Mercury									
16. Nickel									
17. Potassium									
18. Selenium									
19. Silver									
20. Sodium									
21. Thallium									
22. Vanadium									
23. Zinc	–								
Other: _____									
Cyanide									

[1] Initial Calibration Source _____ [2] Continuing Calibration Source _____

[3] Control Limits: Mercury and Tin 80-120; Other Metals 90-110; Cyanide 85-115

[4] Indicate Analytical Method Used: P - ICP; A - Flame AA; F - Furnace AA

Form III

Q. C. Report No. _____

BLANKS

LAB NAME _____ CASE NO. _____

DATE _____ UNITS _____

Matrix _____

Compound	Initial Calibration Blank Value	Continuing Calibration Blank Value 1	2	3	4	Preparation Blank Matrix: 1	Matrix: 2
Metals:							
1. Aluminum							
2. Antimony							
3. Arsenic							
4. Barium							
5. Beryllium							
6. Cadmium							
7. Calcium							
8. Chromium							
9. Cobalt							
10. Copper							
11. Iron							
12. Lead							
13. Magnesium							
14. Manganese							
15. Mercury							
16. Nickel							
17. Potassium							
18. Selenium							
19. Silver							
20. Sodium							
21. Thallium							
22. Vanadium							
23. Zinc	-						
Other:							
Cyanide							

Form IV

Q. C. Report No. _____

ICP INTERFERENCE CHECK SAMPLE

LAB NAME _____ CASE NO. _____

Check Sample I.D. _____

DATE _____ Check Sample Source _____

Units _____

Compound	Control Limits[1] Mean	Control Limits[1] Std. Dev.	True[2]	Initial Observed	Initial %R	Final Observed	Final %R
Metals:							
1. Aluminum							
2. Antimony							
3. Arsenic							
4. Barium							
5. Beryllium							
6. Cadmium							
7. Calcium							
8. Chromium							
9. Cobalt							
10. Copper							
11. Iron							
12. Lead							
13. Magnesium							
14. Manganese							
15. Mercury							
16. Nickel							
17. Potassium							
18. Selenium							
19. Silver							
20. Sodium							
21. Thallium							
22. Vanadium							
23. Zinc							
Other: _____							

[1] Mean value based on n = _____.

[2] True value of EPA ICP Interference Check Sample or contractor standard.

Form V

Q. C. Report No. _____

SPIKE SAMPLE RECOVERY

LAB NAME _____ CASE NO. _____

DATE _____ EPA Sample No. _____

Lab Sample ID No. _____

Units _____

Matrix _____

Compound	Control Limit %R	Spiked Sample Result (SSR)	Sample Result (SR)	Spiked Added (SA)	%R[1]
Metals:					
1. Aluminum	75-125				
2. Antimony	"				
3. Arsenic	"				
4. Barium	"				
5. Beryllium	"				
6. Cadmium	"				
7. Calcium	"				
8. Chromium	"				
9. Cobalt	"				
10. Copper	"				
11. Iron	"				
12. Lead	"				
13. Magnesium	"				
14. Manganese	"				
15. Mercury	"				
16. Nickel	"				
17. Potassium	"				
18. Selenium	"				
19. Silver	"				
20. Sodium	"				
21. Thallium	"				
22. Vanadium	"				
23. Zinc	"				
Other: _____	—				
Cyanide	"				

[1] %R = [(SSR - SR)/SA] x 100

"N" - out of control

"NR" - Not required

Comments: _____

Form VI

Q. C. Report No. _____

DUPLICATES

LAB NAME _____ CASE NO. _____

DATE _____ EPA Sample No. _____

Lab Sample ID No. _____

Units _____

Matrix _____

Compound	Control Limit[1]	Sample(S)	Duplicate(D)	RPD[2]
Metals:				
1. Aluminum				
2. Antimony				
3. Arsenic				
4. Barium				
5. Beryllium				
6. Cadmium				
7. Calcium				
8. Chromium				
9. Cobalt				
10. Copper				
11. Iron				
12. Lead				
13. Magnesium				
14. Manganese				
15. Mercury				
16. Nickel				
17. Potassium				
18. Selenium				
19. Silver				
20. Sodium				
21. Thallium				
22. Vanadium				
23. Zinc				
Other:				
Cyanide				

* Out of Control

[1] To be added at a later date.

[2] RPD = [|S - D|/((S + D)/2)] x 100

NC - Non calculable RPD due to value(s) less than CRDL

Form VII

Q.C. Report No. _____

INSTRUMENT DETECTION LIMITS AND

LABORATORY CONTROL SAMPLE

LAB NAME _____ CASE NO. _____ DATE _____

Compound	Required Detection Limits (CRDL)-ug/l	Instrument Detection Limits (IDL)-ug/l ICP/AA ID#_____	Instrument Detection Limits (IDL)-ug/l Furnace ID#_____	Lab Control Sample ug/L mg/kg (circle one) True	Found	%R
Metals:						
1. Aluminum	200					
2. Antimony	60					
3. Arsenic	10					
4. Barium	200					
5. Beryllium	5					
6. Cadmium	5					
7. Calcium	5000					
8. Chromium	10					
9. Cobalt	50					
10. Copper	25					
11. Iron	100					
12. Lead	5					
13. Magnesium	5000					
14. Manganese	15					
15. Mercury	0.2					
16. Nickel	40					
17. Potassium	5000					
18. Selenium	5					
19. Silver	10					
20. Sodium	5000					
21. Thallium	10					
22. Vanadium	50					
23. Zinc	20					
Other:						
Cyanide	10	NR	NR			

NR - Not required

Form VIII

Q.C. Report No. _____

STANDARD ADDITION RESULTS

LAB NAME _____ CASE NO. _____

DATE _____ UNITS _____

Lab I.D. #	EPA Sample #	Element	0 ADD ABS.	1 ADD		2 ADD		3 ADD		FINAL CON.[2]	r*
				CON.	ABS.[1]	CON.	ABS.[1]	CON.	ABS.[1]		

[1] CON is the concentration added, ABS. is the instrument readout in absorbance or concentration.

[2] Concentration as determined by MSA

*"r" is the correlation coefficient.

+ - correlation coefficient is outside of control window of 0.995.

Form IX

Q. C. Report No. _____

ICP SERIAL DILUTIONS

LAB NAME _____ CASE NO. _____

EPA Sample No. _____

DATE _____ Lab Sample ID No. _____

Units _____

Matrix _____

Compound	Initial Sample Concentration(I)	Serial Dilution[1] Result(S)	% Difference[2]
Metals:			
1. Aluminum			
2. Antimony			
3. Arsenic			
4. Barium			
5. Beryllium			
6. Cadmium			
7. Calcium			
8. Chromium			
9. Cobalt			
10. Copper			
11. Iron			
12. Lead			
13. Magnesium			
14. Manganese			
15. Nickel			
16. Potassium			
17. Selenium			
18. Silver			
19. Sodium			
20. Thallium			
21. Vanadium			
22. Zinc			
Other:			

[1] Diluted sample concentration corrected for 1:4 dilution (see Exhibit D)

[2] Percent Difference = $\frac{|I - S|}{I} \times 100$

NR - Not Required, initial sample concentration less than 10 times IDL

NA - Not Applicable, analyte not determined by ICP

Form X

QC Report No. _____

HOLDING TIMES

LAB NAME _____

DATE _____ CASE NO. _____

Lab ID #	EPA Sample No.	Matrix	Date Received	Mercury Prep Date	Mercury Holding Time (Days)	CN Prep Date	CN Holding Time (Days)

Form XI (Quarterly)
INSTRUMENT DETECTION LIMITS

LAB NAME _____ DATE _____

ICP/Flame AA (Circle One) Model Number_____ Furnace AA Number_____

Element	Wavelength (nm)	CRDL (ug/L)	IDL (ug/L)	Element	Wavelength (nm)	CRDL (ug/L)	IDL (ug/L)
1. Aluminum		200		13. Magnesium		5000	
2. Antimony		60		14. Manganese		15	
3. Arsenic		10		15. Mercury		0.2	
4. Barium		200		16. Nickel		40	
5. Beryllium		5		17. Potassium		5000	
6. Cadmium		5		18. Selenium		5	
7. Calcium		5000		19. Silver		10	
8. Chromium		10		20. Sodium		5000	
9. Cobalt		50		21. Thallium		10	
10. Copper		25		22. Vanadium		50	
11. Iron		100		23. Zinc		20	
12. Lead		5					

Footnotes:
- Indicate the instrument for which the IDL applies with a "P" (for ICP), an "A" (for Flame AA), or an "F" (for Furnace AA) behind the IDL value.
- Indicate elements commonly run with background correction (AA) with a "B" behind the analytical wavelength.
- If more than one ICP/Flame or Furnace AA is used, submit separate Forms XI-XIII for each instrument.

COMMENTS: _____

_____

_____

_____

Lab Manager _____

Form XII (Quarterly)

ICP Interelement Correction Factors

LABORATORY ITAS-Knoxville ICP Model Number _____

DATE _____

Analyte	Analyte Wavelength (nm)	Interelement Correction Factors for Al	Ca	Fe	Mg				
1. Antimony									
2. Arsenic									
3. Barium									
4. Beryllium									
5. Cadmium									
6. Chromium									
7. Cobalt									
8. Copper									
9. Lead									
10. Manganese									
11. Mercury									
12. Nickel									
13. Potassium									
14. Selenium									
15. Silver									
16. Sodium									
17. Thallium									
18. Vanadium	–								
19. Zinc									

COMMENTS: _____

Lab Manager _____

Form XII (Quarterly) (cont'd)

ICP Interelement Correction Factors

LABORATORY _____ ICP Model Number _____

DATE _____

Analyte	Analyte Wavelength (nm)	Interelement Correction Factors for							
1. Antimony									
2. Arsenic									
3. Barium									
4. Beryllium									
5. Cadmium									
6. Chromium									
7. Cobalt									
8. Copper									
9. Lead									
10. Manganese									
11. Mercury									
12. Nickel									
13. Potassium									
14. Selenium									
15. Silver									
16. Sodium									
17. Thallium									
18. Vanadium									
19. Zinc									

COMMENTS: _____

_____

Lab Manager _____

Form XIII (Quarterly)
ICP Linear Ranges

LAB NAME _____ ICP Model Number _____

DATE _____

Analyte	Integration Time (Seconds)	Concentration (ug/L)	Analyte	Integration Time (Seconds)	Concentration (ug/L)
1. Aluminum			13. Magnesium		
2. Antimony			14. Manganese		
3. Arsenic			15. Mercury		
4. Barium			16. Nickel		
5. Beryllium			17. Potassium		
6. Cadmium			18. Selenium		
7. Calcium			19. Silver		
8. Chromium			20. Sodium		
9. Cobalt			21. Thallium		
10. Copper			22. Vanadium		
11. Iron			23. Zinc		
12. Lead					

Footnotes: • Indicate elements not analyzed by ICP with the notation "NA".

COMMENTS: _____

_____

_____

_____

Lab Manager _____

TABLE I

Element	Methods	Footnote
Aluminum	200.7 CLP-M ICP, (6010) ICP, (7020) AA	a,c
Antimony	204.2 CLP-M GFAAS, 200.7 CLP-M ICP (6010) ICP	a,c,f,g
Arsenic	206.2 CLP-M GFAAS	a,c,b
Barium	200.7 CLP-M ICP, (6010) ICP, (7080) AA	a,c
Beryllium	200.7 CLP-M ICP, (6010) ICP, (7090) AA	a,c
Cadmium	200.7 CLP-M ICP, (6010) ICP, (7130) AA	a,c
Calcium	200.7 CLP-M ICP, (6010) ICP, 215.1 CLP-M	a,c
Chromium	200.7 CLP-M ICP, (6010) ICP, (7190) AA	a,c
Cobalt	200.7 CLP-M ICP, (6010) ICP, (7200) AA	a,c
Copper	200.7 CLP-M ICP, (6010) ICP, (7210) AA	a,c
Iron	200.7 CLP-M ICP, (6010) ICP, (7380) AA	a,c
Lead	239.2 CLP-M GFAAS, 200.7 CLP-M (ICP), (6010), (7420) AA	a,c,d,f
Magnesium	200.7 CLP-M ICP, (6010) ICP, 242.1 CLP-M (AA)	a,c
Manganese	200.7 CLP-M ICP, (6010) ICP, (7460) AA	a,c
Mercury	245.1 CLP-M, 245.5 CLP-M	e
Nickel	200.7 CLP-M ICP, (6010) ICP, (7520) AA	a,c
Potassium	200.7 CLP-M ICP, (6010) ICP, 258.1 CLP-M (AA)	a,c
Selenium	270.2 CLP-M GFAAS	a,b,c
Silver	272.2 CLP-M GFAAS, 200.7 CLP-M ICP, (6010) ICP, (7760) AA	a,c,f,g
Sodium	200.7 CLP-M ICP, (6010) ICP, 273.1 CLP-M AA	a,c
Thallium	279.2 CLP-M GFAAS	a,c
Vanadium	200.7 CLP-M ICP, (6010) ICP, (7910) AA	a,c
Zinc	200.7 CLP-M ICP, (6010) ICP, (7950) AA	a,c

Footnotes:

a = Calibration standards prepared from NBS or commercial high purity stock solutions.
b = Nickel nitrate modifier prepared from the metal.
c = Instrument calibration discussed in separate operating procedure.
d = Lanthanum not currently used in modifier for lead analysis by GFAAS.
e = See operating procedure for mercury calibration and analysis for modifications of CLP procedure.
f = 1% HNO_3 used as modifier.
g = Will analyze by ICP if CRDL's can be met.

TABLE II

SAMPLE PREPARATION

LIQUIDS

Water	100 ml sample
Furance AA	1 ml (1 + 1) HNO_3, 2 ml 30% H_2O_2 heat (not boil), dilute to 100 ml
ICP	2 ml (1 + 1) HNO_3, 10 ml (1 + 1) HCl heat (not boil), dilute to 100 ml
Mercury	Attachment 5 and 5A
Cyanide	Attachment 7

SOLIDS (SOILS, SLUDGES, ETC.)

ICP & AA	1. 1.0 gm sample 2. 10 ml HNO_3 (1:1) 3. Heat; reflux 10 minutes 4. 5 ml con. HNO_3 5. Heat; reflux 30 minutes; cool 6. 2 ml H_2O, 3 ml 30% H_2O_2 7. Warm until reaction complete; add up to 10 ml 30% H_2O_2
ICP & Sb	8. 5 ml 1:1 HCl, 10 ml H_2O 9. Cover; heat 10 minutes 10. Cool, filter, dilute up to 200 ml
Furnace AA	8. Reduce to 2 ml 9. Add 10 M H_2O 10. Heat 11. Cool, filter, dilute to 200 ml

TABLE III

POTENTIAL PROBLEMS WITH SAMPLE

SHIPMENT AND ANALYSIS

- Non-homogeneous/multi-phase water or soil samples: Client will be notified and instructions requested. If separation of received sample portions is chosen, client will pay for additional preparation and analysis.

- Matrices other than water or soil (i.e., rocks, leaves, sticks, oil, etc.): Client will be notified that sample will be processed as a soil with appropriate modifications in reagent and sample aliquots.

- Insufficient volume for analysis requested: Client informed no samples in project will be prepared until this is resolved.

- Broken or leaking samples: Client informed no samples in project prepared until situation resolved.

- Incorrent or incomplete paperwork: Client informed work will proceed with target for completion of paperwork.

- Laboratory receipt of incorrect samples: Client notified no work on samples will begin.

- Laboratory accidents involving samples: Client notified work will stop until cause identified and removed.

- Analytical problems with samples: Client notified and kept informed on progress with problem samples on a weekly basis.

TABLE IV

ELEMENTS DETERMINED BY INDUCTIVELY COUPLED PLASMA EMISSION OR ATOMIC ABSORPTION SPECTROSCOPY

Element	Contract Required Detection Level (µg/L)
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5,000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	5
Magnesium	5,000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5,000
Selenium	5
Silver	10
Sodium	5,000
Thallium	10
Vanadium	50
Zinc	20

TABLE V

INITIAL AND CONTINUING CALIBRATION VERIFICATION

CONTROL LIMITS FOR INORGANIC ANALYSES

Analytical Method	Inorganic Species	Percent of True Value (EPA Set)	
		Low Limit	High Limit
ICP/AA	Metals	90	110
Cold Vapor AA	Mercury	80	120

TABLE VI

SPIKING LEVELS FOR SPIKED SAMPLE ANALYSIS[1]

Element	For ICP/AA (µg/L)		For Furance AA (µg/L)		Other (µg/L)
	Water	Sediment[1]	Water	Sediment[1]	
Aluminum	2,000	*			
Antimony	500	500	100	100	
Arsenic			20	40	
Barium	2,000	2,000			
Beryllium	50	50			
Cadmium	50	50	5	5	
Calcium	*	*			
Chromium	200	200			
Cobalt	500	500			
Copper	250	250			
Iron	1,000	*			
Lead	500	500	20	50	
Magnesium	*	*			
Manganese	200	500			
Mercury	10	10			1
Nickel	400	500			
Potassium	*	*			
Selenium			10	10	
Silver	50	50	10	10	
Sodium	*	*	10	10	
Thallium			50	50	
Vanadium	500	500			
Zinc	200	500			
Cyanide					100

NOTE: Elements without spike levels and not designated with an asterisk should be spiked at appropriate levels.

[1]The levels shown indicate concentrations in the final digestate of the spiked sample (200 mL FV).

*No spike required.

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE:	SOP NO: AV871103R0 DATE INITIATED: 11/03/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 8

Instrument Calibration and Sample Analysis for the Determination of Mercury Using the Cold Vapor Technique by CLP Protocol

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Janet M Jones	Jack L. Hall	11/12/87	Mary E. Tyler	11/12/87

1.0 Purpose

The purpose of this SOP is to document the calibration and analysis of mercury by CLP 7/87 protocol using the cold vapor technique.

2.0 Summary

Liquid samples and calibration standards are digested with potassium permanganate and potassium persulfate in a temperature controlled digestion block. The mercury present is then reduced to the elemental state with stannous chloride and aerated from solution and into an absorption cell using the Instrumentation Laboratories Atomic Vapor Accessory (AVA) Model 440. Absorbance (peak height) is measured as a function of mercury concentration.

3.0 References

This procedure is taken largely from Methods 245.5 CLP-M, 245.1 CLP-M, and 245.2 CLP-M of the Contract Laboratory Protocol, SOW #785. The first two methods describe an analysis scheme using BOD bottles and an air bubbler system for generation of mercury vapor. Method 245.2 CLP-M describes analysis using a Technicon Autoanalyzer.

4.0 Sample Handling and Preservation

See Standard Operating Procedure No. A_860624R0.

5.0 Interferences –

The following discussion was taken from Method 245.2 CLP-M and Method 7470, SW-846, 1986 edition.

5.0 Interferences (continued)

5.1 In addition to inorganic forms of mercury, organic mercury compounds may also be present. These organo-mercury compounds will not respond to the flameless atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercury compounds, including phenyl mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100% recovery when used as the oxidant with these compounds. Therefore, a persulfate oxidation step following the addition of the permanganate has been included to insure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement.

5.2 Some sea waters and wastewaters high in chlorides have shown a positive interference, due to the formation of free chlorine which also absorbs radiation of 253.7 nm. Care must be taken to insure that free chlorine is absent before the mercury is reduced. The preliminary purge provided by the AVA unit is an essential step. Additional permanganate may be needed during the oxidation step for these samples.

5.3 Interference from certain volatile organic materials which will absorb at this wavelength is also possible. A preliminary run under oxidizing conditions, without stannous sulfate, would determine if this type of interference is present.

5.4 Formation of a heavy precipitate, in some wastewaters and effluents, has been reported upon addition of concentrated sulfuric acid. If this is encountered, the problem sample cannot be analyzed by this method.

5.5 Potassium permanganate eliminates possible interferences from sulfide.

6.0 Apparatus

6.1 Atomic Absorption Spectrophotometer (AA): The AA unit must have an open sample presentation area in which to mount the absorption cell. Instrument settings recommended by the particular manufacturers should be followed.

6.2 Instrumentation Laboratories Model 440 Atomic Vapor Accessory (AVA): A Teflon cap and Teflon beakers are used. Argon is used as the purge gas.

6.0 Apparatus (continued)

6.3 Quartz Absorption Cell: 12 cm long, 10 mm in diameter.

6.4 Mercury Hollow Cathode Lamp

6.5 Stripchart Recorder

6.6 60 W Light Bulbs: This may be used to prevent condensation inside the cell. The bulb is positioned to shine on the absorption cell maintaining the air temperature in the cell about 10°C above ambient.

6.7 Technicon BD-40 Heating Unit (Digestion Block): This is maintained at 95°C during analysis.

6.8 75 ml Volumetric Digestion Tubes

7.0 Reagents

7.1 Sulfuric/Nitric acid mixture prepared at 14%/7% v/v with Baker "Instra-Analyzed" or equivalent grades of acids.

7.2 Stannous Chloride, Sodium Chloride, Hydroxylamine Hydrochloride Solution: Prepared by diluting 100 grams of stannous chloride, 60 grams of sodium chloride, 60 grams of hydroxylamine hydrochloride and 83 ml of concentrated hydrochloric acid to one liter with Type I water.

7.3 Mercury Stock Solution, 4 ppm: Prepared from high purity commercial standard solutions. The source for calibration is SPEX and verification is NBS. The diluent is 1% HNO_3.

7.3.1 Mercury working standard - dilute stock 1/10 in 1% HNO_3 to produce 0.4 ppm standard.

7.4 4% potassium permanganate solution prepared by diluting 40 grams potassium permanganate in 1 liter volumetric with Type I water.

7.5 4% persulfate solution prepared by diluting 40 grams potassium persulfate in 1 liter volumetric with Type I water.

8.0 Instrument Setup

8.1 Set up and hollow cathode lamp as indicated in Section 6.0.

8.2 Place atomic vapor accessory (AVA) unit in front of AA and connect external flow meter to air cylinder for purging in between cycles. Attach the argon hose to AVA, making sure that it is locked into place.

8.0 Instrument Setup (continued)

8.3 Place absorption cell securely in burner mount and position the cell using stripchart recorder with AA in flame emission mode to obtain peak transmittance. Make both vertical and horizontal adjustments to cell position.

8.4 Place stannous chloride solution in proper fleaker and flush the solution introduction line using a 10 ml variable pipet.

8.5 Verify that tubing attached to exit side area of absorption cell extends into exhaust hood as mercury vapor is highly toxic.

8.6 Open gas cylinder valves and initiate AVA reaction cycle by pressing start button to flush sample lines and cell. Do this at least two times.

9.0 Calibration and Liquid Sample Analysis

9.1 Preparation of Calibration Standards

9.1.1 At least three replicates of the following concentration levels should be prepared: 0.02 ppm, 0.01 ppm, 0.004 ppm, and calibration blank. Five replicates should be prepared for the 0.02 ppm standard to prevent delay in determining proper scale expansion for analysis.

9.1.2 Place 20 ml of Type I water in a series of 75 ml volumetric digestion tubes that have been detergent cleaned and acid rinsed. Depending on the desired concentration, add 1.0 ml, 0.5 ml or 0.2 ml of the 0.4 ppm working standard to the tubes using automatic pipets.

9.1.3 Add 10 ml of HNO_3/H_2SO_4 mixture, 6 ml of permanganate solution, and 4 ml of persulfate solution to each tube. Place tubes in digestion block and heat for 30 minutes.

9.1.4 While standards are heating, check absorption cell position again. Verify that AVA unit is delivering 3 ml of reductant and that reaction time is one minute.

9.2 Analysis of Calibration Standards

9.2.1 After the 30-minute heating period, empty contents of one tube containing 0.02 ppm standard into the Teflon reaction vessel and attach it to the AVA unit through the modified Teflon cap.

9.0 Calibration and Liquid Sample Analysis (continued)

9.2.1.1 Set the scale expansion on the AA to approximately 5.

9.2.1.2 Set integration time to 1/16 seconds.

9.2.1.3 Place the AA instrument in AB mode and balance analyte and background lamp intensities.

9.2.1.4 Turn on the stripchart recorder.

9.2.2 AVA unit should be set at:

9.2.2.1 Reagent amount 5

9.2.2.2 Reaction time 1

9.2.2.3 LPM air - 3

9.2.2.4 Air flow 0.5 LPM

9.2.3 Initiate the reaction sequence on the AVA unit by pushing the start button.

9.2.3.1 The unit will first purge the sample vessel headspace, transfer lines, and absorption cell with argon.

9.2.3.2 After the initial purge, the stannous chloride solution will be introduced and the sample stirred with a stir bar for one minute.

9.2.3.3 A final argon purge follows the reaction period where the volatilized mercury is swept from the reaction vessel into the absorption cell. Residence time in the absorption cell is short; therefore, peaks appear quickly.

9.2.3.4 Air is kept flowing in the transfer lines at all times.

9.2.3.5 Fine tune the scale expansion.

9.2.4 As analysis of standards begins, begin preparing actual samples for analysis.

9.3 Analysis of Liquid Samples: Liquid samples are to be prepared and analyzed in the same way as the calibration standards, beginning with 20 ml of sample instead of 20 ml of water.

9.0 Calibration and Liquid Sample Analysis (continued)

9.3.1 Place two 20 ml portions of the sample in two separate volumetric tubes. Add reagents as specified under Section 9.1.3.

9.3.2 To one of the tubes, add 0.5 ml of the 0.4 ppm working standard. This single, standard addition spike will be used to monitor sample matrix effects, and is labeled RPT.

9.3.3 Analyze solutions as described in Section 9.2.

9.4 Procedure for Solid Mercury Prep

9.4.1 Refer to SOP NO. A_860619RO for glassware preparation.

9.4.2 Weigh a representative 2.0 gram portion of wet sample and place in a 75 ml volumetric digestion tube. This is a modification of Method 245.5 CLP-M instructions for placing 0.2 grams of sample into a 300 ml BOD bottle.

9.4.3 Add 5 ml of concentrated sulfuric acid and 2.5 ml of concentrated nitric acid. Heat for ten minutes in the digestion block at 95°C. This is a modification of Method 245.5 CLP-M instructions calling for a two-minute heating period using a steam bath.

9.4.4 Add 10 ml of Type I water. Allow solution to cool, then carefully add 15 ml of 4% $KMnO_4$ solution and 8 ml of 4% $K_2S_2O_8$ solution. Return tube to digestion block and heat for an additional 30 minutes. This is a modification of Method 245.5 CLP-M which indicates that 50 ml of Type I water should be added and the 30-minute digestion carried out on a steam bath.

9.4.5 After allowing the sample to cool, transfer all of sample to a 200 ml volumetric flask and bring to volume with Type I water. Extracts should be analyzed no later than 48 hours following preparation. This is a modification of Method 245.5 CLP-M instruction which continues with sample treatment and analysis preceding in the same BOD bottle.

9.5 Analysis of Solid Sample Preparation Extracts

9.5.1 Place two 20 ml portions of the extract in separate 75 ml volumetric tubes.

9.5.2 Add 20 ml of water to each tube.

9.0 Calibration and Liquid Sample Analysis (continued)

9.5.3 For RPT, add 0.5 ml of the 0.4 ppm working standard to one tube.

9.5.4 Place in digestion block for 30 minutes. than analyze as described in Section 9.2.

10.0 Calculations

10.1 Sample concentrations will be quantified using a calibration curve obtained from least squares fit of all data points for standards.

10.1.1 Plot all points on peak height versus concentration curve and visually inspect for linearity through all points.

10.1.2 Depending on cell setup, the curve may not be linear through the 0.02 ppm standard. In this case, apply fit to first three standards.

10.1.3 Complete nonlinear region of curve using average of replicates for top and midrange standards as guides, and read values in this region directly from the curve.

10.2 Final sample concentrations will reflect correction for single standard addition result, preparation dilution, and any run dilutions. As the reagent blank must be less than 0.0002 ppm for analysis to proceed, samples are not corrected for it.

11.0 Quality Control

11.1 Calibration curves must be composed of a minimum of three standards and a blank. Each standard must be run at least 3 times and an average value plotted.

11.2 Prepare a 0.004 ppm standard from NBS by pipetting 0.2 ml of 0.4 ppm intermediate into 20 ml of DI water to be used as calibration verification. Calibration verification must be performed at a frequency of 10%.

11.2.1 The observed value must fall within $\pm$ 20% of the true value or calibration must be repeated.

11.2.2 Samples analyzed prior to calibration verification failure and after last successful verification must be reanalyzed.

11.3 One duplicate per project per matrix type is analyzed at a frequency of 20% within the specific project sample batch.

11.0 Quality Control (continued)

11.4 One spike per project per matrix type is analyzed at a frequency of 20% within the specific project sample batch. The spike concentration will be 0.001 ppm for liquid and soil samples. Spike cannot be seen in samples where concentration exceeds 0.001 ppm Hg.

11.4.1 The spike is prepared as follows:

11.4.1.1 Water - pipet 0.05 ml of the 0.4 ppm working standard into the reaction flask containing 20 ml of sample.

$$\frac{0.05 \text{ ml} \times 0.4 \text{ µg/ml}}{20 \text{ ml}} = 0.001 \text{ µg/ml}$$

11.4.1.2 Soil - pipet 0.5 ml of the 0.4 ppm working standard into the 200 ml volumetric prior to sample prep.

$$\frac{0.5 \text{ ml} \times 0.4 \text{ µg/ml}}{200 \text{ ml}} = 0.001 \text{ µg/ml}$$

11.5 The reagent blank value must be less than 0.0002 ppm before sample analysis can begin. The reagent blank will be analyzed throughout the analysis run at a frequency of one per ten samples.

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE:	SOP NO: AG871103R0 DATE INITIATED: 11/03/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 6
Hazardous Substance List Metals Analysis by Graphite Furnace Atomic Absorption Spectroscopy (CLP SOW 787)	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Janet M Jones	Jack R. Hall	11/12/87	Mary Gryder	11/12/87

1.0 Purpose

Included in this operating procedure for sample analysis are an introduction, a current description of operating parameters for IL 951/655 AA/GFAAS and troubleshooting guides. Specific conditions have been included to better illustrate the analysis process. These may change as experience dictates. The analysis protocol follows the CLP SOW 787 and is limited to As, Se, Pb, and Tl determinations.

2.0 Introduction

2.1 General description of procedure: Samples and standards are injected in one to two ml portions onto the inner surface of a graphite cuvette. The sample is then subjected to a predetermined temperature program in such a way that it is progressively dried, charred, and finally atomized. The form or matrix in which the analyte exists can seriously affect the analysis. Matrix modification is employed to reduce or eliminate interferences and to put the analyte in the optimum chemical form for atomization. Sample preparation can be considered part of the modification process. Figure 1, taken from Instrumentation Laboratory's Atomic Absorption Methods Manual, Volume 2, illustrates the difference between GFAAS and AA signals.

2.2 Matrix modifiers:

2.2.1 Selenium and arsenic: 1% (v/v) nitric acid, 200 ppm nickel.

2.2.2 Antimony: 0.5% (v/v) nitric acid, 40 ppm nickel.

2.2.3 Silver, cadmium, lead, chromium, and thallium: 1% (v/v) nitric acid.

2.2.4 May be changed as type of samples or experience dictates. Analysis of a standard reference material will be used to gauge the effectiveness of changes.

2.0 Introduction (continued)

2.3 Cuvette type: Instrument specific - I.L.

2.3.1 Selenium, arsenic, silver, antimony, cadmium, and chromium: Delayed atomization cuvette that has been pyrolytically coated.

2.3.2 Lead and thallium: Delayed atomization cuvette that has not been pyrolytically coated.

2.4 Calibration Standards:

2.4.1 Selenium, arsenic:

Stock solution - 10 ppm - prepared once per month.

Calibration standards (prepared at run time):
200 ppb, 100 ppb, 40 ppb, 20 ppb, 10 ppb, 5 ppb, blank

2.4.2 Lead, thallium:

Stock solution - 2.0 ppm - prepared at run time.

Calibration standards (prepared at run time):
40 ppb, 20 ppb, 10 ppb, 5 ppb, blank

3.0 Sample and Standard Analysis Scheme:

3.1 Standards: 1 ml standard + 1 ml deionized water.

3.1.1 Standards are prepared in the matrix modifier solution.

3.1.2 The matrix modifier solution is used as the calibration blank.

3.2 Samples: 1 ml sample + 1 ml matrix modifier.

3.3 Sample single standard addition 1 ml sample + 1 ml of 2X CRDL standard.

3.4 All samples are initially analyzed using the single standard addition procedure. A known quantity of analyte is added to a second portion of sample as indicated in Section 3.3 and the resulting mixture analyzed. A recovery factor can then be determined which is used to adjust the sample concentration for matrix effects.

3.5 Follow CLP 787 analysis scheme for flame/furnace (Figure 1).

4.0 General Operating Instructions: 951/655/254

4.1 Turn cool flow on. Temp set @ 22°C.

4.2 Turn 951 to operate mode.

4.3 Depress recall. Select element to be analyzed and type in element #, depress Enter. "Menu" for desired element is then displayed.

4.4 Select and install hollow cathode lamp. Turn current to lamp on and set at Nor. Opt. current as stated on lamp.

4.5 Set band width to achieve optimal signal. Set high voltage to achieve optimal signal.

4.6 Depress AC, depress Mode, select mode of operation. Type in #, Enter select channel & mode of operation. Type in #, Enter. Select element - type in #, Enter. Select scale expand, Enter. Type in # Enter. Depress Enter @ statistics prompt . Depress Enter @ D_2 warm up prompt.

4.7 Remove furnace face plate and install new sensor and cuvette - replace face plate.

4.8 Turn on argon cylinder 40 psig. Turn on atomizer 655.

4.9 Turn on autosampler 254.

4.10 Check seal around door on furnace.

4.11 Depress door button on 254 and unlock jet and align properly. Depress door button to close door.

4.12 Unlock temp set and set temperature to read 22°C. Lock temp set.

4.13 Set 655/254 for sensor conditioning conditions.

4.14 Change dri-rite & glass wool (in nebulizer tubing).

4.15 Turn "door" control on 254 to reading of approximately 8.

4.16 Depress single and turn door control slowly until door opens.

4.17 Allow furnace to condition temp sensor.

4.18 After sensor is conditioned, turn 254/655 off. Allow to sit for about 5 minutes.

4.19 Turn 655/254 on reset temp as in step #12..

4.0 General Operating Instructions: 951/655/254 (continued)

4.20 Depress AC/AL on 951. To turn Bkgd current up, check lamp balance by moving HC/D_2 switch back and forth while observing signal on energy meter.

Use of filter may be necessary to balance lamps.

4.21 Select program card for desired element and set conditions on 655/254.

4.22 Turn repeat to 5 and select solution for condition cuvette.

4.23 Repeat steps 15 and 16.

4.24 After cycle complete, turn repeat to 1.

4.25 Analyze the calibration standard with the highest concentration according to the analysis scheme in Section 3.5 and observe the shape of the peak. The mode of operation should be peak height.

4.26 Continue running top standards until the peak tip appears slightly rounded, and the peak sides are symmetric. If after at least five injections ...

4.26.1 ... the peaks are too sharp - atomization temperature may be too high

4.26.2 ...the peaks contain side bumps or bulges - the dry and pyrolyze steps may need adjusting.

4.26.3 ... the peak tops are blunt and peaks broad - the atomization temperature may be too low.

4.26.4 ... trailing appears to right of peak - the cuvette surface may have degraded to the point where cuvette should be replaced.

4.27 Analyze a calibration blank. If a peak appears, either the atomization time or temperature may be too low. If memory persists,—rule out transport contamination by aspirating 1% nitric acid solutions between sample injections. This is necessary for lead analysis. If this fails to correct the problem, replace the furnace cuvette.

5.0 Calibration

Beginning with Blk, analyze three of each of the calibration standards as given in Section 1.4. Calculate the average value for each standard. When the response range (R), from largest to smallest value per standard, divided by the average value (A) per standard is greater than 0.15, the steps given in Section 9 for sensitivity check should be followed. If precision cannot be improved, replace the cuvette.

$\frac{R}{A}$ < 0.15 precision criteria

If precision is acceptable, plot the average values, as well as the individual readings for each standard. Draw a smooth curve through the average values. The graph will be used during sample analysis to monitor the recovery factor. Concentration values should be read from the graph and the values noted on the run chart or calculation worksheet.

6.0 Analysis

6.1 Analyze the initial calibration verification standard and calculate its concentration. Its value must be within $\pm$ ten percent of the true value or the instrument must be recalibrated.

6.2 When calibration verification is complete, begin running samples. See Figure 2 for example run log.

7.0 Temperature Sensor: Instrument Specific - I.L. 655

The temperature sensor should be changed daily and should be checked when the temperature profile is erratic or whenever the cuvette is replaced. The new sensor must be conditioned using the temperature program designed for this purpose.

8.0 Cuvette Lifetime

8.1 Generally, cuvettes should be changed after eight hours of operation or a drop of greater than twenty percent in sensitivity during an analysis.

To restore sensitivity, try the following steps:

8.1.1 Analyze at least three more mid-range calibration standards. This may help to clean the cuvette.

8.1.2 Check sample jet position to verify that it is positioned centrally over the cuvette injection hole.

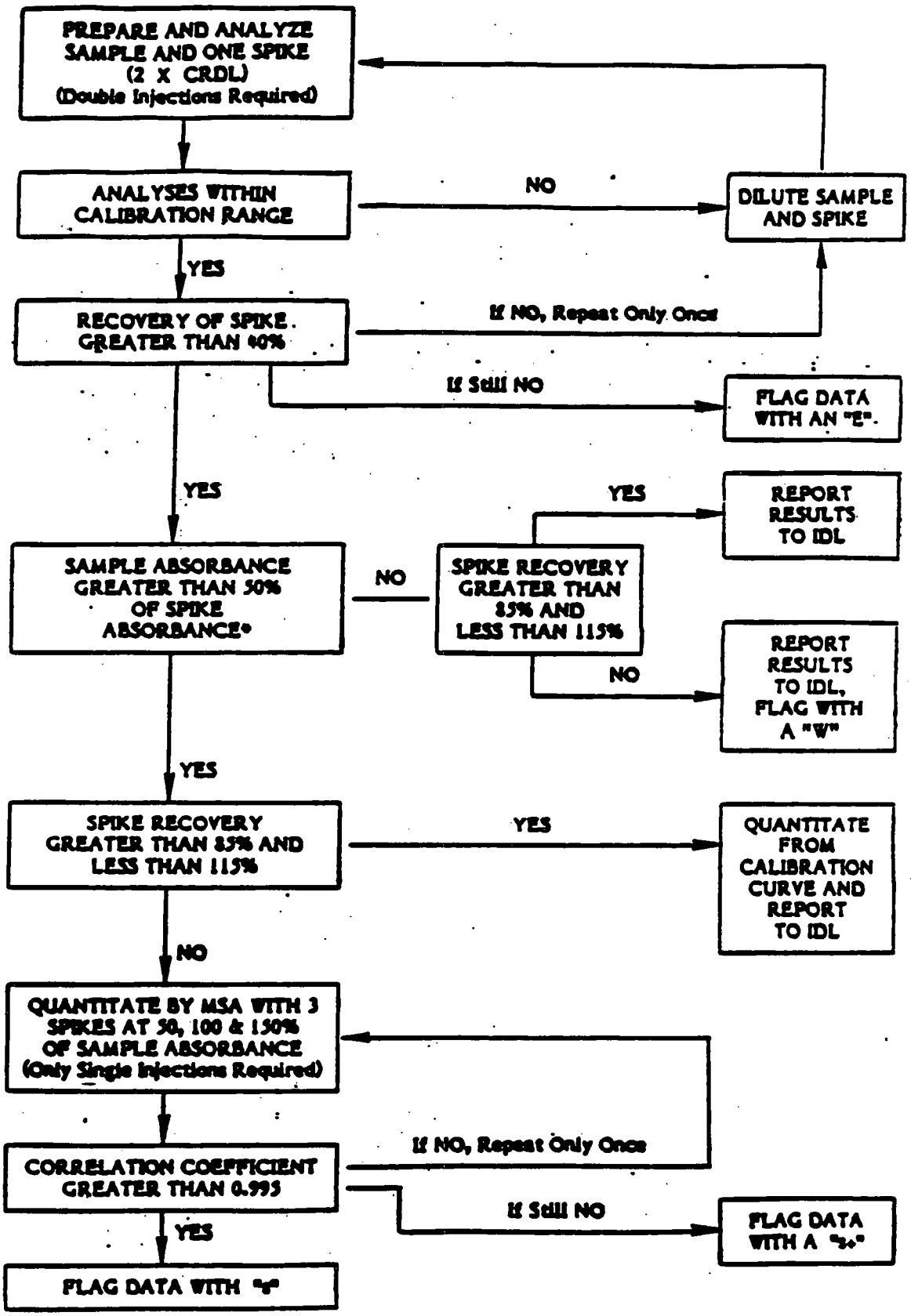
8.0 Cuvette Lifetime (continued)

8.1.3 Monitor temperature profile for the atomization step on several sample injections. A variation greater than 100 degrees between peak temperatures for the injections could indicate a problem with the temperature sensor (in the I.L. Model 655 furnace). Normally, a weakened temperature sensor will produce problems with precision long before the large drop in sensitivity. If the sensor is replaced, it must be conditioned and the instrument recalibrated before analysis can continue.

8.1.4 Replace the dry tube glass wool and absorbant. Moisture may have compacted the absorbant to the point that sample up-take has been affected.

8.2 Samples with high concentrations of interferents may dictate frequent cuvette changes.

Figure 1.
FURNACE ATOMIC ABSORPTION ANALYSIS SCHEME 787 CLP SOW



*Spike absorbance defined as (absorbance of spike sample) minus (absorbance of the sample).

Figure 2

GFAA RUN SUMMARY

1. Calibrate
   - STD1 - BLK
   - STD2 - CRDL
   - STD3 - 2XCRDL
   - STD4
   - STD5

2. Calculate average & plot curve

3. ICV 90-110% criteria
   - If correct value is obtained with RF < 85% or >115%, rerun once to verify repeatability.
   - If incorrect values obtained, try dilution.
   - Do not rerun more than 2 times. If correct value not obtained, recalibrate.

4. ICB
   - <IDL report IDLU
   - >IDL and <CRDL report Value B
   - >CRDL terminate run

5. PB & RPT @ <CRDL
   - If RF is <85% or >115%, rerun once; if still out, terminate run.
   - If above CRDL, then lowest sample must be 10 times the blank concentration or all samples must be reprepped - terminate run.

6. LCS & RPT 80-120% criteria
   - RF 85-115%
   - If correct value not obtained, terminate run

7. Samples - duplicate injections - follow Decision Tree, Figure 1
   - S1 + RPT (2 shots)
     S1 + RPT (2 shots)
   - S2 + RPT (2 shots)
     S2 + RPT (2 shots)
   - S3 - MSA (4 shots)

8. CCV (same as #3)

9. CCB (same as #4)

10. Samples
    - Continue with same scheme as in #7
      10 shots between QC samples
    - MSA counts as 4 shots
    - Sample plus RPT counts as 2 shots

11. End run with QC
    - CCV
    - CB

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: Reporting Hazardous Substance List (HSL) Analysis - Metals, Contract Laboratory Protocol	SOP NO: A 871104R0 DATE INITIATED: 11/03/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 11

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Janet M Jones	Jack R Hall	11/12/87	Mary E Tyler	11-12-87

1.0 Purpose

Taken from the Contract Laboratory Protocol Statement of Work (SOW) #787 (July 1987) this Standard Operating Procedure addresses the handling of HSL analysis requests for metals from sample preparation through sample analysis and data package presentation. Data package forms and parameters presented herein reflect current useage and may change in both content and number with subsequent SOW revisions. This procedure will address the following items: 1) Current data package forms; 2) General sample preparation scheme; 3) Current HSL metals list and methods; 4) Data package contents; 5) Provisions for problems; and, 6) Quality assurance and quality control (QA/QC) requirements for SOW 787.

2.0 HSL Metals List and Methods of Analysis

The methods appear in order of priority for useage with those in parentheses representing method numbers from the September 1986 edition of SW-846. Footnotes appear when additional information is required. The following qualifiers appear to further identify methods: (ICP) Inductively Coupled Plasma, (AA) Direct Aspiration - Flame, (GFAA) Graphite Furnace, (CVAA) Cold Vapor AA - mercury analysis.

3.0 Overview of Sample Preparation

Table II presents the summarized sample preparation scheme. Refer to the individual operating procedures for more detailed descriptions of preparation.

4.0 Sample Data Package Contents

4.1 A completed data package will include the following elements:

- Case narrative
- Cover page - inorganic analysis data package
- Sample results on Form I
- Completed contractual QC Forms II through XIII
- Copies of ICP, GFAAS, Hg digestion logs or comparable worksheets
- Analytical raw data
- Copies of traffic reports and Chain-of-Custody forms

4.2 Blank forms are attached.

4.3 Comments

4.3.1 Result Forms: Header Information

(Sample) U.S. EPA - CLP

COVER PAGE - INORGANIC ANALYSES DATA PACKAGE

Lab Name: _____ Contract: _____
Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
SOW No.: _____

This information is required on every EPA CLP package.

4.3.2 Commercial CLP (Non-EPA) Packages: Header Information

(Sample) U.S. EPA - CLP

COVER PAGE - INORGANIC ANALYSES DATA PACKAGE

Lab Name: ITASK Contract: _____
Lab Code: _____ Case No.: ABC 12345 SAS No.: _____ SDG No.: XYZ
SOW No.: 787

4.0 Sample Data Package Contents (continued)

Use the following spaces for commercial CLP packages:

LAB NAME: ITASK
CASE NO.: (Project Code)
SDG NO.:
SOW NO.: 787
CONTRACT: (use client contract number if applicable)

4.3.3 Forms XI through XIII are generated quarterly for instrument parameter verification.

4.3.4 Analytical raw data includes all information needed to reconstruct sample life from preparation to report.

5.0 Potential Problems and Provisions for Dealing with Them

5.1 Instrument malfunction: If the ICP unit malfunctions, those elements affected will be analyzed for by AAS.

5.2 Table III presents a list of potential problems and how they will be dealt with. Attempts will be made to provide flexibility in all areas.

5.3 Solid samples will not be mixed and pulverized. Reasonable attempts will be made to obtain a homogeneous aliquot without destroying sample integrity.

5.4 Problems will be documented in the case narrative and/or nonconformance memos.

6.0 QA/QC Requirements

This section outlines the minimum QA/QC operations necessary to satisfy the analytical requirements of the contract.

1. Instrument Calibration
2. Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)
3. CRDL Standards for AA (CRA) and ICP (CRI)
4. Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses
5. ICP Interference Check Sample (ICS) Analyses
6. Spike Sample Analysis (S)
7. Duplicate Sample Analysis (D)
8. Laboratory Control Sample (LCS) Analysis
9. ICP Serial Dilution Analysis (L)

6.0 QA/QC Requirements (continued)

10. Instrument Detection Limit (IDL) Determination
11. Interelement Corrections for ICP (ICP)
12. Linear Range Analysis (LRA)
13. Furance AA QC Analyses

6.1 Instrument Calibration

6.1.1 Instruments must be calibrated each time the instrument is set up.

6.1.2 AA Systems

- Blank + 3 calibration standards
- One standard must be at the CRDL (except for Hg)

6.1.3 ICP Systems

- Follow instrument manufacturer's recommended procedures (minimum: blank + 1 standard)
- To verify linearity near the CRDL, a 2X CRDL standard must be analyzed at the beginning and end of each sample analysis run, or a minimum of twice per 8 hour working shift, whichever is more frequent (for all ICP elements except Al, Ba, Ca, Fe, Mg, Na, and K).

6.2 Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)

6.2.1 The accuracy of the initial instrument calibration must be verified and documented for every analyte by the analysis of Initial Calibration Verification Solutions (ICV).

6.2.2 If an ICV is not available from EPA or where a certified solution of an analyte is not available from any source, analyses shall be conducted on an independent standard at a concentration other than that used for calibration, but within the calibration range.

6.2.3 Independent standard: Standard composed of analytes from a different source than those used in the standards for the initial instrument calibration.

NOTE: Generally, SPEX is used for calibration and NBS for verification when available.

6.0 QA/QC Requirements (continued)

6.2.4 The ICV must be run at each wavelength used for analysis.

6.2.5 The ICV must fall within the specified control limits (Table V).

6.2.6 ICV results must be recorded on QC Form II.

6.2.7 Continuing Calibration Verification (CCV) must be performed for each analyte at a frequency of 10% or every two hours during an analysis run, whichever is more frequent.

6.2.8 CCV must also be analyzed for each analyte at the beginning and end of the analysis run.

6.2.9 The same continuing calibration standard must be used throughout the analysis run for a particular case. This standard may be the same as the ICV.

6.2.10 One of the following standards must be used for continuing calibration verification:

1. EPA solution
2. NBS SRM
3. Contractor prepared solution

6.2.11 If CCV results exceed the specified control limits (Table V), the instrument must be recalibrated and the preceding 10 samples reanalyzed for the analytes affected.

6.2.12 CCV results must be recorded on Form II.

6.3 CRDL Standards

6.3.1 To verify linearity near the CRDL, the Contractor must analyze a standard at two times the CRDL or two times the IDL, whichever is greater, at the beginning and end of each sample analysis run, or a minimum of twice per eight hour-working shift, whichever is more frequent, but not before Initial Calibration Verification. This standard must be run by ICP for every wavelength used for analysis, except those for Al, Ba, Ca, Fe, Mg, Na, and K. Separate standards are run for GFAA analysis for As, Se, Pb, Tl, and CVAA analysis for Hg.

6.4 Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses

6.4.1 Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) Analyses

6.0 QA/QC Requirements (continued)

6.4.1.1 A calibration blank must be analyzed at each wavelength used for analysis immediately after every initial and continuing calibration verification at a frequency of 10% or every two hours during the run, whichever is more frequent. The blank must be analyzed at the beginning of the run and after the last analytical sample. The results for the calibration blanks shall be recorded on Form III-IN for ICP, AA, and cyanide analyses, as indicated.

6.4.2 Preparation Blank (PB) Analysis

6.4.2.1 At least one preparation blank (or reagent blank) consisting of deionized distilled water processed through each sample preparation and analysis procedure must be prepared and analyzed with every Sample Delivery Group or with each batch* of samples digested, whichever is more frequent.

*A group of samples prepared at the same time.

6.5 ICP Interference Check Sample Analysis

Frequency: Beginning and end of each sample analysis run (minimum 2x/8 hours)

6.5.1 ICP Interference Check Samples (ICS) supplied by EPA (EMSL-LV).

6.5.2 ICS results must fall within the control limit of ± 20% of the EPA supplied true value for the analytes included in the ICS. Otherwise, terminate the analysis, correct the problem, recalibrate, reverify the calibration, and reanalyze the samples.

6.5.3 If EPA ICS is not available, an independent ICS must be prepared with the interferent and analyte concentrations at the levels specified in Table VII.

6.5.4 For the independent standard, the mean value and standard deviation must be established by initially analyzing the ICS at least 5x repetitively for each parameter listed on Form IV.

6.5.5 Results of the contractor prepared ICS must fall within the control limit of ± 20% of the established mean value.

6.5.6 ICS result must be recorded on Form IV.

6.0 QA/QC Requirements (continued)

6.6 Spiked Sample Analysis

6.6.1 Predigestion spike (matrix spike)

6.6.2 At least one spiked sample analysis must be performed on each group of samples of a similar matrix type for each case of samples or for each 20 samples received, whichever is more frequent.

6.6.3 Samples identified as field blanks cannot be used for spiked sample analysis.

6.6.4 Analyte spike levels are specified in Table VI.

6.6.5 If spike recovery is not written within the limits of 75- 125%, all data associated with that spike must be flagged "N" (exception: when sample concentration is 4x spike concentration).

6.6.6 $$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

where: SSR = spiked sample result
SR = sample result (where SR < IDL, use SR = 0)
SA = spike added

6.6.7 Spiked sample results must be reported on Form V.

6.6.8 If two analytical methods are used to obtain the reported values for the same element for a case of samples, spike samples must be run by each method used.

6.7 Duplicate Sample Analysis

6.7.1 At least one duplicate sample must be analyzed from each group of samples of a similar matrix type for each case of samples or for each 20 samples received, whichever is more frequent.

6.7.2 Samples identified as field blanks cannot be used for duplicate sample analysis.

6.7.3 If two analytical methods (i.e., ICP, AA) are used to obtain the reported values for the same element for a case of samples, duplicate samples must be run by each method used.

6.0 QA/QC Requirements (continued)

6.7.4 $RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$

where: RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (duplicate)

6.7.5 Duplicate sample results must be reported on Form VI.

6.7.6 Control limits: ± 20% RPD for sample results > 5x CRDL
± CRDL for sample results < 5x CRDL
± CRDL for one result > 5x CRDL, the other < 5x CRDL
if either result < CRDL, RPD is "N.C."

6.7.7 Flag all associated results for RPD's which exceed the control

6.8 Laboratory Control Sample (LCS) Analysis

The LCS must be analyzed for each analyte using the same methods employed for samples (preparation and analysis).

6.8.1 Aqueous (LCSW)

6.8.1.1 One LCSW must be prepared and analyzed for every 20 samples received, or for each batch of samples digested, whichever is more frequent.

6.8.1.2 For Hg, LCSW is not required.

6.8.1.3 Results must be reported on QC Form VII.

6.8.1.4 If results (%R) exceed control limits of 80-120%, analyses must be terminated, the problem corrected, and the samples associated with that LCS reanalyzed.

6.8.2 Solid LCS (LCSS)

6.8.2.1 The availability and use of a LCSS is limited to EPA projects only. An alternate source for the LCSS is being sought.

6.0 QA/QC Requirements (continued)

6.8.2.2 Currently, this laboratory is using a liquid concentrate standard reference material with certified values to verify solid sample preparation. It is prepared with samples at a frequency of one per twenty samples per project. The certified values are converted to mg/kg using 200 ml/1 g and reported as LCSS. This may change depending on SOW revisions and availability of solid material with control limits.

6.9 ICP Serial Dilution Analysis

6.9.1 Must be performed on each group of samples of a similar matrix type (i.e., water, soil) for each case of samples or for each 20 samples received, whichever is more frequent.

6.9.2 Samples identified as field blanks cannot be used for serial dilution analysis.

6.9.3 An analysis of a 1:5 dilution must agree within 10% of the original determination on the undiluted sample when the analyte concentration is minimally a factor of 10x IDL after dilution.

6.9.4 If the original analyte value is not at least 10 times the IDL, that element will not be used in the percent difference determination.

6.9.5 If the dilution analysis is not within 10%, the data must be flagged with an "E".

6.9.6 Serial dilution results must be reported on QC Report Form IX.

6.10 Quarterly Verification of Instrument Parameters

6.10.1 Instrument Detection Limit (IDL) Determination

6.10.1.1 IDL's must be determined prior to the analysis of any field samples under the contract and at least quarterly for each instrument.

6.10.1.2 IDL's must meet the Contract Required Detection Limits (CRDL) specified in Table IV.

6.10.1.3 IDL's are three times the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration 3-5 times IDL, with seven consecutive measurements per day.

6.0 QA/QC Requirements (continued)

6.10.1.4 QC Report Form XI and the documentation for IDL determinations must be submitted as part of the data package.

6.10.1.5 For each case, IDL's must be reported on QC Report Form VII.

6.10.1.6 If multiple instruments of the same type are used for the analysis of an element within a case, the highest IDL for that instrument type must be reported on the QC Report Form VII for that case.

6.11 Interelement Correction Factors

6.11.1 Determine as per instrument manufacturer's instructions.

6.11.2 Report correction factors on QC Report Form XII.

6.12 Linear Range Analysis

6.12.1 Linear range verification check standard must be analyzed and reported quarterly for each element on QC Form XII.

6.12.2 Analytically determined concentration of this standard must be written ± 5% of the true value.

6.12.3 The concentration of the standard run defines the upper limit of the ICP linear range beyond which results cannot be reported without dilution.

6.12.4 When an analyte concentration exceeds the linear range, reanalysis of the prepared sample, after appropriate dilution, is required.

6.13 Furnace Atomic Absorption QC Analysis

6.13.1 Duplicate Injections

6.13.1.1 Required for all furnace analyses except during full MSA.

6.13.1.2 Raw data must contain both readings, the average value and the RSD or CV average result must be reported on Form I.

6.13.1.3 For concentrations > CRDL, duplicate injection readings must agree within 20% RSD or CV or the sample must be rerun once.

6.0 QA/QC Requirements (continued)

6.13.1.4 If after the third injection the readings are still out, flag the value with a "M" on Form I.

6.13.2 Analytical Spikes (Post-Digest)

6.13.2.1 All furnace analyses for each sample requires at least a single analytical spike.

6.13.2.2 Analytical spikes are not required on predigest spike sample.

6.13.2.3 Percentage recovery determines how the sample will be quantitated (refer to Figure 1).

6.13.3 Method of Standard Additions (MSA) Requirements

6.13.3.1 Data must be within linear range as determined by the calibration curve.

6.13.3.2 The original sample and the three spikes must be analyzed consecutively.

6.13.3.3 Only single injections are required.

6.13.3.4 Spikes should be prepared such that:

Spike 1 is ≈ 50% of the sample absorbance
Spike 2 is ≈ 100% of the sample absorbance
Spike 3 is ≈ 150% of the sample absorbance

6.13.3.5 Raw data must include slope, intercept and correlation coefficient (r).

6.13.3.6 MSA results must be reported on Form VIII.

6.13.3.7 Results obtained by MSA must be flagged "s" on Form I.

6.13.3.8 If $r < 0.995$, the MSA must be repeated once. If 2nd r is still < 0.995, then flag Form I result with "+".

6.13.3.9 See Figure 1 for flow chart of furance analysis scheme.

TABLE I

Element	Methods	Footnote
Aluminum	200.7 CLP-M ICP, (6010) ICP, (7020) AA	a,c
Antimony	200.7 CLP-M ICP (6010) ICP	a,c
Arsenic	206.2 CLP-M GFAAS, 200.7 CLP-M ICP, (6010) ICP	a,c,b
Barium	200.7 CLP-M ICP, (6010) ICP, (7080) AA	a,c
Beryllium	200.7 CLP-M ICP, (6010) ICP, (7090) AA	a,c
Cadmium	200.7 CLP-M ICP, (6010) ICP, (7130) AA	a,c
Calcium	200.7 CLP-M ICP, (6010) ICP, 215.1 CLP-M	a,c
Chromium	200.7 CLP-M ICP, (6010) ICP, (7190) AA	a,c
Cobalt	200.7 CLP-M ICP, (6010) ICP, (7200) AA	a,c
Copper	200.7 CLP-M ICP, (6010) ICP, (7210) AA	a,c
Iron	200.7 CLP-M ICP, (6010) ICP, (7380) AA	a,c
Lead	239.2 CLP-M GFAAS, 200.7 CLP-M (ICP), (6010), (7420) AA	a,c,d,f
Magnesium	200.7 CLP-M ICP, (6010) ICP, 242.1 CLP-M (AA)	a,c
Manganese	200.7 CLP-M ICP, (6010) ICP, (7460) AA	a,c
Mercury	245.1 CLP-M, 245.5 CLP-M	e
Nickel	200.7 CLP-M ICP, (6010) ICP, (7520) AA	a,c
Potassium	200.7 CLP-M ICP, (6010) ICP, 258.1 CLP-M (AA)	a,c
Selenium	270.2 CLP-M GFAAS, 200.7 CLP-M ICP, (6010) ICP	a,b,c
Silver	200.7 CLP-M ICP, (6010) ICP, (7760) AA	a,c
Sodium	200.7 CLP-M ICP, (6010) ICP, 273.1 CLP-M AA	a,c
Thallium	279.2 CLP-M GFAAS, 200.7 CLP-M ICP, (6010) ICP	a,c
Vanadium	200.7 CLP-M ICP, (6010) ICP, (7910) AA	a,c
Zinc	200.7 CLP-M ICP, (6010) ICP, (7950) AA	a,c

Footnotes:

a = Calibration standards prepared from SPEX or commercial high purity stock solutions.
b = Nickel nitrate modifier prepared from the metal.
c = Instrument calibration discussed in separate operating procedure.
d = Lanthanum not currently used in modifier for lead analysis by GFAAS.
e = See operating procedure for mercury calibration and analysis for modifications of CLP procedure.
f = 1% HNO_3 used as modifier.

TABLE II

SAMPLE PREPARATION

LIQUIDS

Water	100 ml sample
Furance AA	1 ml (1 + 1) HNO_3, 2 ml 30% H_2O_2 heat (not boil), dilute to 100 ml
ICP	2 ml (1 + 1) HNO_3, 10 ml (1 + 1) HCl heat (not boil), dilute to 100 ml
Mercury	See SOP

SOLIDS (SOILS, SLUDGES, ETC.)

ICP & AA

1. 1.0 gm sample
2. 10 ml HNO_3 (1:1)
3. Heat; reflux 10 minutes
4. 5 ml con. HNO_3
5. Heat; reflux 30 minutes; cool
6. 2 ml H_2O, 3 ml 30% H_2O_2
7. Warm until reaction complete; add up to 10 ml 30% H_2O_2
8. 5 ml 1:1 HCl, 10 ml H_2O
9. Cover; heat 10 minutes
10. Cool, filter, dilute up to 200 ml

Furnace AA

11. Cool, filter, dilute to 200 ml

TABLE III

POTENTIAL PROBLEMS WITH SAMPLE

SHIPMENT AND ANALYSIS

- Non-homogeneous/multi-phase water or soil samples: Client will be notified and instructions requested. If separation of received sample portions is chosen, client will pay for additional preparation and analysis.

- Matrices other than water or soil (i.e., rocks, leaves, sticks, oil, etc.): Client will be notified that sample will be processed as a soil with appropriate modifications in reagent and sample aliquots.

- Insufficient volume for analysis requested: Client informed no samples in project will be prepared until this is resolved.

- Broken or leaking samples: Client informed no samples in project prepared until situation resolved.

- Incorrent or incomplete paperwork: Client informed work will proceed with target for completion of paperwork.

- Laboratory receipt of incorrect samples: Client notified no work on samples will begin.

- Laboratory accidents involving samples: Client notified work will stop until cause identified and removed.

- Analytical problems with samples: Client notified and kept informed on progress with problem samples.

TABLE IV

ELEMENTS DETERMINED BY INDUCTIVELY COUPLED PLASMA EMISSION OR ATOMIC ABSORPTION SPECTROSCOPY

Element	Contract Required Detection Level (µg/L)
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5,000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	5
Magnesium	5,000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5,000
Selenium	5
Silver	10
Sodium	5,000
Thallium	10
Vanadium	50
Zinc	20

TABLE V

INITIAL AND CONTINUING CALIBRATION VERIFICATION

CONTROL LIMITS FOR INORGANIC ANALYSES

Analytical Method	Inorganic Species	Percent of True Value (EPA Set)	
		Low Limit	High Limit
ICP/AA	Metals	90	110
Cold Vapor AA	Mercury	80	120

TABLE VI

SPIKING LEVELS FOR SPIKED SAMPLE ANALYSIS[1]

Element	For ICP/AA (µg/L)		For Furance AA (µg/L)		Other (µg/L)
	Water	Sediment[1]	Water	Sediment[1]	
Aluminum	2,000	*			
Antimony	500	500	100	100	
Arsenic	2,000	2,000	40	40	
Barium	2,000	2,000			
Beryllium	50	50			
Cadmium	50	50	5	5	
Calcium	*	*			
Chromium	200	200			
Cobalt	500	500			
Copper	250	250			
Iron	1,000	*			
Lead	500	500	20	20	
Magnesium	*	*			
Manganese	500	500			
Mercury	10	10			
Nickel	500	500			1
Potassium	*	*			
Selenium	2,000	2,000	10	10	
Silver	50	50			
Sodium	*	*			
Thallium	2,000	2,000	50	50	
Vanadium	500	500			
Zinc	500	500			
Cyanide					100

NOTE: Elements without spike levels and not designated with an asterisk should be spiked at appropriate levels.

[1]The levels shown indicate concentrations in the final digestate of the spiked sample (200 mL FV).

*No spike required.

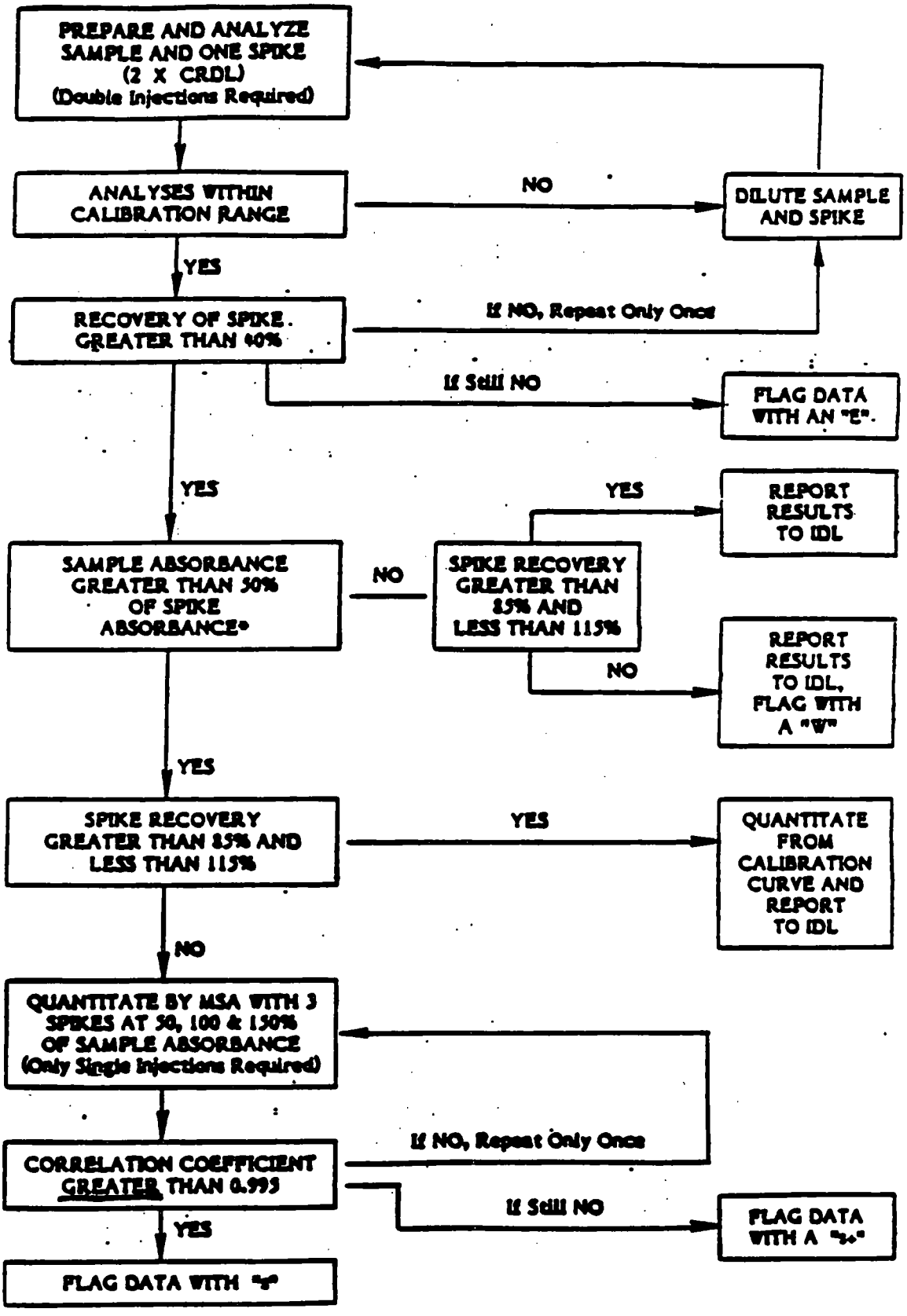
TABLE VII

INTERFERENT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR

ICP INTERFERENCE CHECK SAMPLE

Analytes	(mg/L)	Interferents	(mg/L)
Ag	1.0		
Ba	0.5	Al	500
Be	0.5	Ca	500
Cd	1.0	Fe	200
Co	0.5	Mg	500
Cr	0.5		
Cu	0.5		
Mn	0.5		
Ni	1.0		
Pb	1.0		
V	0.5		
Zn	1.0		

Figure 1.
FURNACE ATOMIC ABSORPTION ANALYSIS SCHEME



*Spike absorbance defined as (absorbance of spike sample) minus (absorbance of the sample).

U.S. EPA - CLP

COVER PAGE - INORGANIC ANALYSES DATA PACKAGE

b Name: ______________________ Contract: __________

Lab Code: ______ Case No.: ______ SAS No.: ______ SDG No.: ______

SOW No.: ______

EPA Sample No.	Lab Sample ID.

Were ICP interelement corrections applied? Yes/No ___

Were ICP background corrections applied? Yes/No ___
If yes-were raw data generated before application of background corrections? Yes/No ___

Comments:

____

____

____

____

Release of the data contained in this hardcopy data package and in the computer-readable data submitted on floppy diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Lab Manager: ______________________

Date: __________

1

INORGANIC ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix (soil/water): _____ Lab Sample ID: _____

Level (low/med): _____ Date Received: _____

% Solids: _____

Concentration Units (ug/L or mg/kg dry weight): _____

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum				
7440-36-0	Antimony				
7440-38-2	Arsenic				
7440-39-3	Barium				
7440-41-7	Beryllium				
7440-43-9	Cadmium				
7440-70-2	Calcium				
7440-47-3	Chromium				
7440-48-4	Cobalt				
7440-50-8	Copper				
7439-89-6	Iron				
7439-92-1	Lead				
7439-95-4	Magnesium				
7439-96-5	Manganese				
7439-97-6	Mercury				
7440-02-0	Nickel				
7440-09-7	Potassium				
7782-49-2	Selenium				
7440-22-4	Silver				
7440-23-5	Sodium				
7440-28-0	Thallium				
7440-62-2	Vanadium				
7440-66-6	Zinc				
	Cyanide				

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:

2A

INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: ITASK Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Initial Calibration Source: NBS

Continuing Calibration Source: NBS

Concentration Units: ug/L

Analyte	Initial Calibration True	Initial Calibration Found	Initial Calibration %R(1)	Continuing Calibration True	Continuing Calibration Found	Continuing Calibration %R(1)	Continuing Calibration Found	Continuing Calibration %R(1)	M
Aluminum	40,000			40,000					a
Antimony	4,000			4,000					a
Arsenic	4,000			4,000					a
Barium	4,000			4,000					a
Beryllium	4,000			4,000					a
Cadmium	4,000			4,000					a
Calcium	40,000			40,000					a
Chromium	4,000			4,000					a
Cobalt	4,000			4,000					a
Copper	4,000			4,000					a
Iron	40,000			40,000					a
Lead	4,000			4,000					b
Magnesium	40,000			40,000					a
Manganese	4,000			4,000					a
Mercury	N/A			N/A					b
Nickel	4,000			4,000					b
Potassium	40,000			40,000					a
Selenium	4,000			4,000					b
Silver	4,000			4,000					b
Sodium	4,000			4,000					a
Thallium	4,000			4,000					b
Vanadium	4,000			4,000					a
Zinc	4,000			4,000					a
Cyanide									

(1) Control Limits: Mercury 80-120; Other Metals 90-110; Cyanide 85-115

NOTE: The above values are for ICAP only

2B
CRDL STANDARD FOR AA AND ICP

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

AA CRDL Standard Source: _____

ICP CRDL Standard Source: _____

Concentration Units: ug/L

Analyte	CRDL Standard for AA			CRDL Standard for ICP				
	True	Found	%R	True	Initial Found	%R	Final Found	%R
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								

3
BLANKS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Preparation Blank Matrix (soil/water): _____

Preparation Blank Concentration Units (ug/L or mg/kg): _____

Analyte	Initial Calib. Blank (ug/L)	C	Continuing Calibration Blank (ug/L) 1	C	2	C	3	C	Prepa-ration Blank	C	M
Aluminum											
Antimony											
Arsenic											
Barium											
Beryllium											
Cadmium											
Calcium											
Chromium											
Cobalt											
Copper											
Iron											
Lead											
Magnesium											
Manganese											
Mercury											
Nickel											
Potassium											
Selenium											
Silver											
Sodium											
Thallium											
Vanadium											
Zinc											
Cyanide											

4
ICP INTERFERENCE CHECK SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ ICS Source: _____

Concentration Units: ug/L

Analyte	True Sol. A	True Sol. AB	Initial Found Sol. A	Initial Found Sol. AB	Initial Found %R	Final Found Sol. A	Final Found Sol. AB	Final Found %R
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc		-						

U.S. EPA - CLP

5A
SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

.b Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix (soil/water): _____ Level (low/med): _____

Concentration Units (ug/L or mg/kg dry weight): _____

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
ron									
ead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

U.S. EPA - CLP

5B

POST DIGEST SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix (soil/water): _____ Level (low/med): _____

Concentration Units: ug/L

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

6 DUPLICATES

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix (soil/water): _____ Level (low/med): _____

% Solids for Sample: _____ % Solids for Duplicate: _____

Concentration Units (ug/L or mg/kg dry weight): _____

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Cyanide								

U.S. EPA - CLP

7

LABORATORY CONTROL SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Solid LCS Source: _____

Aqueous LCS Source: _____

Analyte	Aqueous (ug/L) True	Found	%R	Solid (mg/kg) True	Found	C	Limits		%R
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

8

STANDARD ADDITION RESULTS

b Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Concentration Units: ug/L

EPA Sample No.	An	Dil	0 ADD ABS	1 ADD CON	1 ADD ABS	2 ADD CON	2 ADD ABS	3 ADD CON	3 ADD ABS	Final Conc.	r	Q

FORM VIII - IN 7/87

9
ICP SERIAL DILUTIONS

EPA SAMPLE NO.

ab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix (soil/water): _____ Level (low/med): _____

Concentration Units: ug/L

Analyte	Initial Sample Result (I)	C	Serial Dilution Result (S)	C	% Difference	Q	M
Aluminum							
Antimony							
Arsenic							
Barium							
Beryllium							
Cadmium							
Calcium							
Chromium							
Cobalt							
Copper							
Iron							
Lead							
Magnesium							
Manganese							
Mercury							
Nickel							
Potassium							
Selenium							
Silver							
Sodium							
Thallium							
Vanadium							
Zinc							

U.S. EPA - CLP

10
HOLDING TIMES

Lab Name: ______________________ Contract: __________

Lab Code: ______ Case No.: _____ SAS No.: ______ SDG No.: ______

EPA Sample No.	Matrix	Date Received	Mercury Prep Date	Mercury Holding Time	Cyanide Prep Date	Cyanide Holding Time

11

INSTRUMENT DETECTION LIMITS (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ Date: _____

Flame AA ID Number: _____

Furnace AA ID Number: _____

Analyte	Wave-length (nm)	Back-ground	CRDL (ug/L)	IDL (ug/L)	M
Aluminum			200		
Antimony			60		
Arsenic			10		
Barium			200		
Beryllium			5		
Cadmium			5		
Calcium			5000		
Chromium			10		
Cobalt			50		
Copper			25		
Iron			100		
Lead			5		
Magnesium			5000		
Manganese			15		
Mercury			0.2		
Nickel			40		
Potassium			5000		
Selenium			5		
Silver			10		
Sodium			5000		
Thallium			10		
Vanadium			50		
Zinc			20		

Comments:

12A
ICP INTERELEMENT CORRECTION FACTORS (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ Date: _____

Analyte	Wave-length (nm)	Interelement Correction Factors for: Al	Ca	Fe	Mg	_____
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Mercury						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						
_____						

Comments:

_____

_____

_____

_____

U.S. EPA - CLP

12B

ICP INTERELEMENT CORRECTION FACTORS (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ Date: _____

Analyte	Wave-length (nm)	Interelement Correction Factors for:				
		_____	_____	_____	_____	_____
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Mercury						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						
_____						

Comments:

_____

_____

_____

_____

13
ICP LINEAR RANGES (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ Date: _____

Analyte	Integ. Time (Sec.)	Concentration (ug/L)	M
Aluminum			
Antimony			
Arsenic			
Barium			
Beryllium			
Cadmium			
Calcium			
Chromium			
Cobalt			
Copper			
Iron			
Lead			
Magnesium			
Manganese			
Mercury			
Nickel			
Potassium			
Selenium			
Silver			
Sodium			
Thallium			
Vanadium			
Zinc			

Comments:

_____

_____

_____

_____

STANDARD OPERATING PROCEDURE

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE:	SOP NO: A 871105R0 DATE INITIATED: 11/06/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 4
Preparation of Solid Samples for Metals - Contract Laboratory Protocol (SOW 787)	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
James M Jones	Jack A Hall	11/12/87	Mary E Taylor	11/12/87

1.0 Purpose

Taken from the Contract Laboratory Protocol Statement of Work #787 (July 1987), procedure describes the preparation of solid samples for analysis by inductively coupled plasma (ICP), graphite furnace atomic absorption spectroscopy (GFAAS), and flame atomic absorption spectroscopy (AAS). (See SOP AV871103R0 for mercury sample prep.)

2.0 Procedure

2.1 Screening and Documentation

2.1.1 Chain-of-Custody: Samples are removed from temporary storage after the appropriate checkout notebook has been signed. Project specific Chain-of-Custody forms follow the samples through the preparation phase. See Figure 1C.

2.1.2 Screening: Prior to preparation, the sample pH is checked and the value recorded on the project specific sample tracking form. At this time, information regarding preparation type and client identification is recorded in the central sample preparation logbook, as is the date of preparation. See Figure 1 for sample tracking sheet.

2.1.3 Documentation: In addition to the Chain-of-Custody forms and the central preparation logbook, samples are tracked on a tracking sheet which contains information such as weight of sample used, description of sample, and observation of sample before, during, and after prep. Copies are made of this completed form and passed with the sample to the analysis lab. See Figures 1a, 1b, and 1d for sample tracking sheet and logbook page.

2.0 Procedure (continued)

2.2 Reagents

2.2.1 ASTM Type I deionized water

2.2.2 Baker "Instra-analyzed" acids or equivalent

2.2.3 Hydrogen peroxide - reagent grade

2.3 Sample Preparation

2.3.1 Glassware preparation: See SOP No. A_860619R1

2.3.2 GFAAS Preparation (As, Se, Pb, Tl only): Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 gms) a 1.0 to 1.5 gm portion of sample and transfer to a beaker.

2.3.2.1 Add 10 ml of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass. Heat the sample to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5 ml of concentrated HNO_3, replace the watch glass, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 ml while maintaining a covering of solution over the bottom of the beaker.

2.3.2.2 After the second reflux step has been completed and the sample has cooled, add 2 ml of Type I water and 3 ml of 30% hydrogen peroxide (H_2O_2). Return the beaker to the hot plate for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.

2.3.2.3 Continue to add 30% H_2O_2 in 1 ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 ml 30% H_2O_2).

2.3.2.4 If the sample is being prepared for the furnace analysis of As, Pb, Se, Tl, continue heating the acid-peroxide digestate until the volume has been reduced to approximately 2 ml, add 10 ml of Type I water, and warm the mixture. After cooling, filter through Whatman No. 2 filter paper and dilute to 100 ml with Type I water (or centrifuge the sample). The diluted digestate solution contains approximately 2% (v/v) HNO_3. Dilute the digestate 1:1 (200 ml final volume) with deionized water. For analysis, withdraw aliquots of appropriate volume, and add any required reagent or matrix modifier. The sample is now ready for analysis.

2.0 Procedure (continued)

2.3.3 ICP/AAS sample preparation: Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 gms) a 1.0 to 1.5 gm portion of sample and transfer to a beaker.

2.3.3.1 Add 10 ml of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass. Heat the sample to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5 ml of concentrated HNO_3, replace the watch glass, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 ml while maintaining a covering of solution over the bottom of the beaker.

2.3.3.2 After the second reflux step has been completed and the sample has cooled, add 2 ml of Type I water and 3 ml of 30% hydrogen peroxide (H_2O_2). Return the beaker to the hot plate for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.

2.3.3.3 Continue to add 30% H_2O_2 in 1 ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 ml 30% H_2O_2).

2.3.3.4 If the sample is being prepared for the flame AA or ICP analysis of Al, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Ag, Na, Tl, V, and Zn, add 5 ml of 1:1 HCl and 10 ml of Type II water, return the covered beaker to the hot plate, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 2 filter paper (or equivalent) and dilute to 100 ml with Type II water. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v) HNO_3. Dilute the digestate 1:1 (200 ml final volume) with the deionized water. The sample is now ready for analysis.

3.0 Quality Control

3.1 Laboratory Control Sample (LCS): Prepare with the samples at a frequency of one per twenty samples, this standard reference material is used to monitor the effectiveness of sample preparation. Current sources for the LCS are the EPA, the NBS, and the ERA.

3.1.1 Due to the unavailability of a solid LCS for projects other than for the EPA, the liquid LCS is prepared using the solid prep method and converted to mg/kg using 200 ml/1 g factor.

3.2 Preparation Blanks: Preparation blanks are prepared concurrently with each set of samples at a minimum frequency of one per twenty samples each time preparation is initiated.

3.3 Preparation Duplicates: Preparation duplicates are prepared at a minimum frequency of one per twenty samples per project.

3.4 Predigest Spikes: Predigest spikes are prepared at a minimum frequency of one per twenty samples per project. (See Figure 2 for spiking levels.)

3.5 A QC sample initiation form is used to list samples by number and project code. When the 20th sample is reached, another form is started with QC prepped on the 1st sample on the sheet. (See Figure 3.)

3.6 Any sample/preparation nonconformances are indicated on a nonconformance memo and distributed to the group supervisor, QC Coordinator, Lab Manager, and project file. See Figure 1e.

FIGURE 1a

SAMPLE TRACKING
METALS

MS/MSD Spike Std. Conc. _____
Volume Added _____
Spike Std. Date _____
Sample No. Spiked _____

Project Code _____
Date Prepped _____ By _____
Commercial _____ CLP _____
Type of Prep _____

Sample ID No.	Vol/Wgt AA	GF	Hg	pH	Preserve Y or N	% Solids	Matrix/ Description	Observations

ITAS-K-A_013RI

FIGURE 1b

IT ANALYTICAL SERVICES - KNOXVILLE

SAMPLE PREPARATION LOGBOOK - METALS

Sample No.	Prep Type	Weight	Prep Date of LCS and Inclusive Sample Range	Comments

FIGURE 1c

Project Code _____
EPA Case # _____
Sample No. Range _____

INTERNAL CHAIN-OF-CUSTODY FORM - METALS
IT Analytical Services-Knoxville

Sample No.	Date	Extract* Type or Original Sample?	Moved From	Moved To	Reason	Signature

* O - Original Sample
WH - Water, Hydrochloric Acid Finish
WN - Water, Nitric Acid Finish
DH - Dirt (Soil, Sediment, etc.), Hydrochloric Acid Finish
DN - Dirt (Soil, Sediment, etc.), Nitric Acid Finish

FIGURE 1d

PROJECT PREP WORKSHEET FOR GROUP LEADERS PAGE 1

METALS

PROJECT CODE=EVR24282 DUE DATE=05/18/87 DATE ISSUED=05/17/87 10:54

SAMPLE(S)	TY	R?	DUE DATE	PREP	ID	ANALYST	DATE
DD1293\DD1297	01	E		PA11	Hg Preservation	_____	__/__/__
	01	E		PA12	CLP-Furn.-H2O	_____	__/__/__
	01	E		PA14	CLP-I/F/G-H2O	_____	__/__/__
DD1308	31	E		PA04 701	Hg in Sediment	_____	__/__/__
	31	E		PA13	CLP-Furn.-Soil	_____	__/__/__
	31	E		PA15	CLP-I/F/G-Soil	_____	__/__/__
DD1309/DD1311	31	E		PA04 701	Hg in Sediment	_____	__/__/__
	31	E		PA13	CLP-Furn.-Soil	_____	__/__/__
	31	E		PA15	CLP-I/F/G-Soil	_____	__/__/__
DD1312	11	E		PA04 701	Hg in Sediment	_____	__/__/__
	11	E		PA13	CLP-Furn.-Soil	_____	__/__/__
	11	E		PA15	CLP-I/F/G-Soil	_____	__/__/__
DD1313/DD1318	11	E		PA04 701	Hg in Sediment	_____	__/__/__
	11	E		PA13	CLP-Furn.-Soil	_____	__/__/__
	11	E		PA15	CLP-I/F/G-Soil	_____	__/__/__

INSTRUCTIONS: USE CLP PROTOCOL
DD1293-97: LIQUID
DD1296 - SPLIT OF '93 DD1297 - SPIKE OF '93
THERE MAY BE OTHER QC - LET ME KNOW AFTER CHECKING BOTTLES

SPECIAL QC : DD1308-11: SOLID SAMPLES
DD1312-18: OIL SAMPLES - PREP AS SOLIDS

PREP-NOTES :

PREPPED BY: _____, __/__/__ APPROVED BY: _____, __/__/__

NONCONFORMANCE MEMO
ITAS-KNOXVILLE

AA/ICP DATA REVIEW

DATE _____
PROJECT CODE _____
FILED BY _____

SAMPLE NO.(s) _____

NONCONFORMANCE: (Check applicable item(s)):

_____ (1) Method development or modification to include procedures not currently used on a regular basis (requires QA approval). (SPECIFY) _____

_____ (2) Calibration failure: (SPECIFY) _____

_____ (3) Sample identification/dilution error: (SPECIFY) _____

_____ (4) Calculation/transcription error: (SPECIFY) _____
(a) Error discovered before report to client.
(b) Error discovered after report to client.

_____ (5) Matrix spike/duplicate: _____
(a) Not recoverable due to high concentration in original sample.
(b) Not determinable due to possible sample inhomogeneity.
(c) Not determinable due to matrix effects.
(d) % Recovery / % RPD outside prescribed limits.
(e) Other: (SPECIFY) _____

_____ (6) Specified detection limit unobtainable due to: _____
(a) Matrix interferences.
(b) Limited sample volume.
(c) Blank criteria not met.
(d) Other: (SPECIFY) _____

_____ (7) Standard operating procedure not adhered to. (SPECIFY) _____

_____ (8) Holding time exceeded by _____ (days).

_____ (9) Sample received unpreserved.

_____ (10) Other: (SPECIFY) _____

CORRECTIVE ACTION TAKEN (Check applicable item(s)):

_____ (1) Error corrected by analyst. (SPECIFY) _____

_____ (2) Error corrected/resolved by QC Coordinator. (SPECIFY) _____

_____ (3) Situation noted on sample tracking sheet and appropriate lab personnel notified. (SPECIFY) _____

_____ (4) Sample processed "as is".

_____ (5) Sample preserved with _____ and let sit _____ prior to processing.

_____ (6) Samples put "on hold" until further notice.

_____ (7) Spike/standard concentration verified. New solution made if necessary.

_____ (8) Samples reanalyzed.

_____ (9) Samples reprepped and reanalyzed.

_____ (10) Client informed verbally.

_____ (11) Client informed by memo/letter.

_____ (12) Other (SPECIFY): _____

ROUTING

Title	Initials	Date	Check if Corrected
Analyst	_____	_____	_____
Group Supervisor	_____	_____	_____
QC Coordinator (if necessary)	_____	_____	_____
Assistant Lab Manager (if necessary)	_____	_____	_____

ITAS-K-QA016R2

FIGURE 2

CLP SPIKES - SOW 787

ELEMENT	REQ CONC PPM	ML STD NEEDED	STOCK CONC PPM	SPIKE CONC PPM
SOLUTION #1 AA/ICP CLP SOW 787				
Aluminum	2	20	1,000	200
Arsenic	2	20	1,000	200
Barium	2	20	1,000	200
Selenium	2	20	1,000	200
Thallium	2	20	1,000	200
final volume = 100ml				
SOLUTION #2 AA/ICP CLP SOW 787				
Iron	1	10	1,000	100
Antimony	0.5	5	1,000	50
Cobalt	0.5	5	1,000	50
Lead	0.5	5	1,000	50
Manganese	0.5	5	1,000	50
Nickel	0.5	5	1,000	50
Vanadium	0.5	5	1,000	50
Zinc	0.5	5	1,000	50
Copper	0.25	2.5	1,000	25
Chromium	0.2	2	1,000	20
Beryllium	0.05	0.5	1,000	5
Cadmium	0.05	0.5	1,000	5
Silver	0.05	0.5	1,000	5
final volume = 51 ml of standards brought up to 100 ml				
SOLUTION #3 GFAAS CLP SOW 787				
Antimony	0.1	10	1,000	100
Thallium	0.05	5	1,000	50
Arsenic	0.04	4	1,000	40
Lead	0.02	2	1,000	20
Selenium	0.01	1	1,000	10
Cadmium	0.005	0.5	1,000	5
final volume = 22.5 ml of standards brought up to 100 ml				

FOR AA/ICP PREPS:

a. WATER (100 ml final volume) use 1 ml of SOLUTION #1 & 1 ml of SOLUTION #2
b. SOIL (200 ml final volume) use 2 ml of SOLUTION #1 & 2 ml of SOLUTION #2

FOR GFAAS PREPS:

a. WATER (100 ml final volume) use 0.1 ml of SOLUTION #3
b. SOIL (200 ml final volume) use 0.2 ml of SOLUTION #3

MERCURY SPIKES: 0.001 ppm is required

a. Make up a 1ppm Hg standard at the time of analysis by taking 0.05 ml of the 1,000 ppm stock standard and diluting up to 50 ml.
b. For water sample analysis: use 0.02 ml of the 1 ppm standard you made in a. (for 20 ml sample volume)
c. For soil samples: use 0.2 ml of the 1 ppm standard you made in a. (for 200 ml final volume). If you are using 250ml volumetrics for the soil prep: use 0.25 ml of the 1 ppm standard.

FIGURE 3
IT ANALYTICAL SERVICES
QC Sample Initiation Form
AA/ICP

QA/QC Sample ID: _____

Prep Code: _____ QC Type:(2) _____ Date Initiated: _____
Prep Name: _____ Date Completed: _____
Matrix: _____ Sample
Project Code:(1) _____ (Lab) ID: _____ Approved By: _____

Comments:

Prep Date/Analyst		Project Code	Sample ID	Prep/Blk
	1)			
	2)			
	3)			
	4)			
	5)			
	6)			
	7)			
	8)			
	9)			
	10)			
	11)			
	12)			
	13)			
	14)			
	15)			
	16)			
	17)			
	18)			
	19)			
	20)			
	21)			
	22)			

1) In the sample ID column, mark the original sample with an OS.
2) QC Type Designations
   B = Blank
   D = Duplicate
   S = Spike
   R = Reference Material or Standard
   K = Known (stable) Standard

ITAS-K-A_010R0

REQUEST FOR ISSUANCE OF FORMS OR S.O.P.'s

BLOCK 1A
Document Title: Standards Prep Metals
Developed by: KSW - JMJ
Date: 11-3-87

BLOCK 1B
Service Group/Department: AA
Form? _____ SOP? ✓
Revision? 1
Group Supervisor Approval: [signature]
Date: 11-3-87

BLOCK 1C
Technical Director Approval:
Date: _____

BLOCK 2
Management Approval: [signature]
Date: 11/5/87

BLOCK 3
Document No.: AA870204R1
QA Concurrence: [signature]
Date: 11-5-87

BLOCK 4
CPT/Instrument Operator: [signature]
Date: 11-11-87
Disc: 5.25's #16
File Name: 870204R1

BLOCK 5
Original Document(s) to: JMJ
Copies of Document to: RMW, SAM, ARM

INSTRUCTIONS

1) Person(s) developing or revising document should fill out Blocks 1A and 5, attach form to document, and give to Group Leader/Department Coordinator.

2) Group Supervisor should fill out Block 1B, add to Block 5, and give to Technical Director.

3) Technical Director should approve document (Block 1C), add to Block 5 and turn in to ITAS Management responsible for activity.

4) ITAS Management should approve document (Block 2), add to Block 5, and send to QA Department (JMJ).

5) QA Department approves, assigns Document No. (Block 3), and sends to Word Processing or back to Group if form is to be developed on instrument data systems.

6) CPT operator or developer from Group fills out Block 4, disperses document as specified in Block 5, and attaches this form to the original(s) of the final document and sends to QA Files (JMJ), and others as specified in Block 5.

STANDARD OPERATING PROCEDURE

IN-01

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE:	SOP NO: IS851022RO DATE INITIATED: 10/21/85 REVISION NO: 0 DATE REVISED: PAGE 1 of 2
Samples to be Analyzed for Inorganic Parameters Following USEPA Contract Laboratory Program (CLP) Protocol	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Gary M Woody	Jack R Hall	10/23/85	Gary M Woody	10/23/85

1.0 Scope and Applications

This SOP applies to all aqueous and solid samples submitted to IT Corporation by the USEPA, and of the various states, or commercial clients for the analysis for inorganic parameters following CLP protocol and procedures specified in EPA IFB WA-85J839 (SOW 785, July 1985) and subsequent amendments. Such samples are hereafter referred to as "CLP samples".

2.0 Sample Receipt, Preservation, Storage, and Handling

2.1 CLP samples shall be received and logged in following SOP CD-841010RO. It is at this point that samples are checked for proper preservation and chain-of-custody documentation.

2.2 CLP samples or sample fractions requiring refrigerated storage shall be stored following SOP QA841113RO. Refrigerated storage areas are monitored following SOP MA841214RO.

2.3 Sample handling, work assignments, analysis tracking, and internal chain-of-custody procedures given in SOP QA841214RO-1 will be followed. Facility security is maintained per SOP QA841114RO.

2.4 CLP sample holding times will be those specified in EPA IFB WA-85J839 (SOW 785, July 1985) and subsequent amendments.

3.0 Analytical Methods

Analytical methods and procedures specified in EPA IFB WA-85J839 (SOW 785, July 1985) and subsequent amendments are to be used exclusively for CLP samples. In the event that the client desires the analysis for parameters not covered by SOW 785, or for sample matrices not covered by SOW 785, alternate or additional EPA approved methods may be used only after prior written agreement between IT Corporation and the client regarding such methods and the costs of analysis. For the determination of hexavalent chromium, methods 7195 and 3060 from Test Methods for Evaluating Solid Waste (EPA SW-846, second edition) shall be used for aqueous and solid samples, respectively.

4.0 Data Recording and Reporting

4.1 General laboratory data reporting procedures specified in SOP QA841214RO-5 shall be followed.

4.2 Unless there is prior written agreement between IT Corporation and the client to the contrary, the report forms and format to be used shall be those specified by IFB WA-85J839 and subsequent amendments.

5.0 Quality Assurance/Quality Control (QA/QC)

5.1 QA/QC requirements shall be those specified by IFB WA-85J839 and subsequent amendments. QC forms and format to be used shall be those specified by the IFB.

5.2 General internal laboratory QA/QC procedures are further governed by the following SOP'S:

5.2.1 Balance Calibration: QA841214RO-3

5.2.2 Water Purification System Monitoring: QA841214RO-6

5.2.3 Glassware Cleanup for Organic Extractions and Analyses: QA841214RO-2

5.2.4 Laboratory Data Storage Procedures, Gas Chromatograms: GC840523R1

IT CORPORATION

IT ANALYTICAL SERVICES

STEWART LABORATORIES DIVISION

TITLE:	SOP NO: GC850624R0 DATE INITIATED: 06/24/85 REVISION NO: 0 DATE REVISED: PAGE 1 of 43
Analysis of Pesticides and PCB's Under the CLP Contract	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Elizabeth Tawney Mary Deakin	Jack R. Hall	7/10/85	James E. Atkins	

1.0 Purpose

1.1 This SOP details procedures followed by ITAS-Knoxville for the analysis of CLP HSL pesticides and PCB's. The CLP contract is the primary SOP for this analysis.

1.2 Samples and standards are to be chromatographed, calculated and reported according to the CLP contract protocol. Changes to the contract protocol will be implemented as they are made by EPA. This SOP documents ITAS specific additional and/or more detailed procedures for the analysis of HSL pesticides and PCB's. EPA and ITAS forms for calculations and reporting of data are included.

2.0 GC Analysis

2.1 Samples and standards are injected into the GC using an autosampler. A solvent wash is loaded after each standard or sample. If an original undiluted soil extract must be injected, it is injected manually using the autosampler syringe. As samples and standards are loaded into the autosampler trays, the ITAS sample number or standard name is recorded on the GC run log sheet. Fill out all required information on the run log sheet.

2.2 The PE 7500 and the LCI-100 are set up to collect the data following the procedure in the manual for the PE 7500. The setup information is to include the SMO case number, the injection volume, and the instrument ID. The method header information includes the column type. Enter in the required information following the manual instructions.

2.3 The 24-Hour Sequence for Pesticide/PCB Analysis is as follows:

	Sample or Standard
1.	Evaluation standard mix A
2.	Evaluation standard mix B
3.	Evaluation standard mix C
4.	Individual standard mix A
5.	Individual standard mix B
6.	Toxaphene
7.	Tech. chlordane
8.	Aroclors 1016/1260
9.	Aroclor 1221
10.	Aroclor 1232
11.	Aroclor 1242
12.	Aroclor 1248
13.	Aroclor 1254
14.	5 samples *
15.	Evaluation standard mix B
16.	5 samples
17.	Individual standard mix A or B
18.	5 samples
19.	Repeat the above sequence starting with Evaluation standard mix B (step 15 above).
20.	Pesticide/PCB analysis sequence must end with Individual standard mix A or B regardless of number of samples analyzed.

* On the primary analytical column, if aldrin and endrin meet the linearity requirements but DDT does not, then the three DDT linearity standards are substituted for the first three samples.

2.4 After EPA-A and EPA-B mixed standards are run, the GC method is modified to update the component retention times and response factors. Follow instructions in the PE 7500 manual for calibrating and modifying the method. Be sure all single component pesticides are identified correctly and that response factors are correct (d-BHC is usually incorrectly identified).

2.5 As soon as possible, use the PE 7500 computer to calculate linearity, retention time windows, calibration factors and % difference in calibration factors. These computer printouts are given to the quantitation analyst along with the GC chromatograms.

2.6 In cases where the integrator has obviously drawn a baseline incorrectly, the data is reintegrated using the PE 7500 after repositioning the baseline. After reintegration, the chromatogram is replotted and a new report is printed. These chromatograms and reports are part of the GC chromatogram package.

3.0 Running Samples/Standards and Forms to be Filled out by GC Operator

3.1 Pesticide Evaluation Standards Summary (Form VIII)

3.1.1 This form is used to report all of the twenty-four (24) hour requirements during pesticide analysis. Header information is filled in as explained in Section 4.3. Leave Case Number blank. Date of Analysis should include both dates if analysis runs beyond midnight.

3.1.2 Evaluation Standard Mix A, B, and C must be analyzed every 24 hours to check the linearity of the GC system. Calculate and report the Calibration Factor (total peak area/conc (ppm)/ injection volume (µl) for each of the four pesticides (aldrin, endrin, 4,4'-DDT and Dibutylchlorendate) at each concentration level. There is a program to be used to do this calculation on the PE 7500. Calculate and report the percent relative standard deviation (% RSD) for each of the four compounds. The RSD must be less than 10 percent for aldrin, endrin, and dibutylchlorendate. If the % RSD for 4,4'-DDT exceeds 10 percent on the mixed column, run the DEDT standard series as the first three samples of the analysis. Calculate and report on DEDT linearity form the linearity of DDT, DDE, and DDD. If DDT's linearity is greater than 10% RSD in the DEDT series, all three compounds in the standard must be graphed (concentration versus peak area). The samples containing these compounds will have the concentration of each of these 3 compounds read directly off the graph instead of using a response factor.

$$\% \text{ RSD} = \frac{SD}{\bar{x}} \times 100 \qquad \text{Eq. 1.1}$$

where: $SD = \sum_{i=1}^{N} \frac{(x_i - \bar{x})^2}{N-1}$ = std deviation

$\bar{x}$ = mean of initial three Calibration factors (per compound)

3.1.3 After running EPA-A and EPA-B, the chemist or technician running the GC will update the retention times and the response factors of the method being used to collect the data. This person should next use the computer to calculate retention time (RT) windows and calibration factors. This information is then filled in on Form IX. Leave the Case Number blank on the heading and do not fill in the column marked Conf. or Quant. Save the computer printout and staple it to the raw data sheets later on.

3.0 Running Samples/Standards and Forms to be Filled out by GC Operator (continued)

3.1.4 Once the RT windows are established, reevaluate the chromatograms for Evaluation A, B, and C to make sure the peaks for aldrin, endrin, DDT, DDE, DDD, endrin aldehyde, endrin ketone, and dibutylchlorendate fit inside the windows and are correctly labeled in the data report. Then contiue filling out Form VIII.

3.1.5.1 Evaluation Standard Mix B must be analyzed after every ten samples during a twenty-four hour period. Calculate and report the percent breakdown for 4,4'-DDT and/or endrin for the mixed phase GC column (see equation below). Enter results in appropriate columns. Provide the laboratory identification and time of analysis, for each analysis of the Evaluation Standard Mix B. Laboratory ID is the PE 7500 file number or the LCI-100 file number if the PE 7500 is not used. Time of analysis includes date if analysis runs beyond midnight. Time is reported in military time.

Eq. 1.2

$$\text{\% breakdown for 4,4'-DDT} = \frac{\text{Total DDT degradation peak area (DDE + DDD)}}{\text{Total DDT peak area (DDT + DDE + DDD)}} \times 100$$

Eq. 1.3

$$\text{\% breakdown for endrin} = \frac{\text{Total endrin degradation peak areas (endrin aldehyde + endrin ketone)}}{\text{Total endrin peak area (endrin + endrin aldehyde + endrin ketone)}}$$

3.1.5.2 Calculate the percent breakdown for endrin or 4,4'-DDT on the OV-1 column using Equations 1.2 and 1.3. The percent breakdown must not exceed 20 percent for endrin or 4,4'-DDT.

If there is evidence of a peak at the retention time of 4,4'-DDD/endrin aldehyde (which coelute on the OV-1 GC column), calculate a combined percent breakdown for endrin/4,4'-DDT using Equation 1.4. The combined degradation must not exceed 20 percent.

3.0 Running Samples/Standards and Forms to be Filled out by GC Operator

Eq. 1.4

Combined % Breakdown =

Total endrin/DDT degradation peak area (DDD, DDE, endrin aldehyde, endrin ketone)

Total endrin/DDT peak area (endrin, endrin aldehyde, endrin ketone, DDD, DDE, DDT)

3.1.6 Every standard, sample, and blank must contain the surrogate dibutylchlorendate at the specified level for both water and/or soil/sediment samples. The retention time shift for dibutylchlorendate on packed columns must not exceed 2 percent (0.3 percent for capillary columns) difference (%D) between the initial standard (Evaluation Standard Mix A) and any sample analyzed during the 12-hour time period. Calculate and report the percent difference (%D) for all samples, standards and blanks. Fill in Laboratory ID and time of analysis for each sample and blank. Laboratory ID is file number from PE 7500 or from LCI-100 if PE 7500 is not used and the ITAS sample number. On first sample run after midnight, write in new date as well as time in the Time of Analysis blank. Time is reported in military time. SMO sample number is left blank until case is completed.

Eq. 1.5

$$\% \text{ Difference} = \frac{RT_i - RT_s}{RT_i} \times 100$$

where RT_i = absolute retention time of dibutylchlorendate in the initial standard (Evaluation Mixture A).

RT_s = absolute retention time of dibutylchlorendate in the sample, blank, or any standard analyzed after Evaluation Mixture A.

3.1.7 Form VIII is required for each twenty-four (24) hour period, for each GC system and for each GC column used to analyze HSL Pesticide/PCB's.

3.1.8 Form VIII is the responsibility of the chemist running the GC's. It should be completed before the chromatograms are given to the person doing the calculations.

Pesticide Evaluation Standards Summary

Case No. _____ Laboratory _____

Contract No. _____ GC Column _____

Date of Analysis _____ Instrument ID _____

EVALUATION CHECK FOR LINEARITY

LABORATORY ID				
PESTICIDE	CALIBRATION FACTOR EVAL. MIX A	CALIBRATION FACTOR EVAL. MIX B	CALIBRATION FACTOR EVAL. MIX C	% RSD (≤10%)
ALDRIN				
ENDRIN				
4,4'-DDT(1)				
DIBUTYL CHLORENDATE				

EVALUATION CHECK FOR 4,4'-DDT/ENDRIN BREAKDOWN

	PERCENT BREAKDOWN EXPRESSED AS TOTAL DEGRADATION			
	EVAL. MIX B	EVAL. MIX B	EVAL. MIX B	EVAL. MIX B
LABORATORY ID				
TIME OF ANALYSIS				
ENDRIN				
4,4'-DDT				
COMBINED(2)				

EVALUATION OF RETENTION TIME SHIFT FOR DIBUTYLCHLORENDATE

SMO SAMPLE NO.	LAB ID	TIME OF ANALYSIS	PERCENT DIFF.	SMO SAMPLE NO.	LAB ID	TIME OF ANALYSIS	PERCENT DIFF.

(1) SEE EXHIBIT E, SECTION 7.5.4

(2) SEE EXHIBIT E, SECTION 7.3.1.2.2.1

1/85

CASE NO. _____ LABORATORY ITAS-Knoxville

CONTRACT NO. EPA 68-01-7025 GC COLUMN _____

DATE OF ANALYSIS _____ INSTRUMENT ID _____

EVALUATION CHECK FOR LINEARITY

Laboratory ID				
Pesticide	DEDT 1 Calibration Factor	DEDT 2 Calibration Factor	DEDT 3 Calibration Factor	% RSD < 10%
4,4'-DDE*				
4,4'-DDD*				
4,4'-DDT*				
Dibutylchlorendate				

* When % RSD is greater than 10%, standards must be graphed and concentration in sample extracts read directly off graph.

3.2 Pesticide/PCB Standards Summary (Form IX)

3.2.1 This form is used to monitor the variation in the Calibration Factor for each pesticide standard during the twelve (12) hour period.

3.2.2 Complete the header information including Laboratory Name and Contract Number. This form is required for each twelve hours, for each GC system and for each GC column used to analyze HSL Pesticides/PCB's.

3.2.3 Individual Standard Mix A or B must be analyzed at or near the beginning of a twelve hour period and again at the end. Enter the date of analysis and time of analysis (in military time) in the appropriate spaces for each of the two analyses. Report the retention time (RT) and retention time window for each compound in Individual Standard Mix A or B (retention time window calculated by computer). Calculate the Calibration Factor for each compound using Equation 1.5 and report results on the appropriate column.

Eq. 1.5

$$\text{Calibration Factor} = \frac{\text{Total peak area of a Standard}}{\text{Conc of std (ppm) x inj vol (µl)}}$$

At the end of the 12 hour period calculate and report the percent difference in the Calibration Factor for each pesticide using Equation 1.6.

Eq. 1.6

$$\text{Percent Difference (\%D)} = \frac{Ab_1 - Ab_2}{Ab_1} \times 100$$

where,
Ab_1 = Calibration Factor from the initial standard
Ab_2 = Calibration Factor from the standard at the end of the 12 hour period.

The percent difference between the individual Calibration Factors for each compound in the pesticide standard may vary no more than 15 percent during a twelve hour quantitation run, nor more than 20 percent during a twelve hour confirmation run.

3.2 Pesticide/PCB Standards Summary (Form IX) (continued)

3.2.4 The laboratory is required to provide alpha and gamma-chlordane data only for "weathered" chlordane samples.

3.2.5 Do not fill in column labeled Conf. or Quant. until case is completely calculated.

3.2.6 As the chromatograms are running, check each sample for peaks matching the window of a standard(s).

3.2.6.1 If the response for any of these compounds is 100% or less of full scale, the extract is ready for confirmation and quantitation.

3.2.6.2 If the response for any compound is greater than 100% of full scale, dilute the extract so that the peak will be between 50 and 100% full scale and reanalyze on the packed column. Use this dilution also for confirmation and quantitation.

3.2.6.3 For dilution > 10 fold. Also inject an aliquot of a dilution 10 fold more concentrated to determine if other compounds of interest are present at lower concentrations.

3.2.6.4 Computer reproductions of chromatograms manipulated to ensure all peaks are on scale over a 100 fold range are an accepted substitute. However, this can be no greater than a 100 fold range. This is to prevent retention time shifts by column or detector overload. Linearity must be demonstrated over the 100 fold range using higher concentrations of the evaluation mixture.

3.2.6.5 Adjust the baseline before each run begins to keep baseline from going off scale in the negative direction.

4.0 Instructions For Labeling Chromatograms

4.1 Pesticide standard chromatograms and data system printouts for all standards to include:

- Evaluation Standard Mix A
- Evaluation Standard Mix B
- Evaluation Standard Mix C

EPIC EACB ST DARDS SUMMARY

Case [illegible] _____ Laboratory _____ _____

Contract No. _____ GC Column _____ GC Instrument ID _____

	DATE OF ANALYSIS _____ TIME OF ANALYSIS _____ LABORATORY ID _____				DATE OF ANALYSIS _____ TIME OF ANALYSIS _____ LABORATORY ID _____			
COMPOUND	RT	RETENTION TIME WINDOW	CALIBRATION FACTOR	CONF. OR QUANT.	RT	CALIBRATION FACTOR	CONF. OR QUANT.	PERCENT DIFF. **
alpha-BHC								
beta-BHC								
delta-BHC								
gamma-BHC								
Heptachlor								
Aldrin								
Heptachlor Epoxide								
Endosulfan I								
Dieldrin								
4,4'-DDE								
Endrin								
Endosulfan II								
4,4'-DDD								
Endrin Aldehyde								
Endosulfan Sulfate								
4,4'-DDT								
Methoxychlor								
Endrin Ketone								
Tech. Chlordane								
alpha-Chlordane*								
gamma-Chlordane*								
Toxaphene								
Aroclor - 1016								
Aroclor - 1221								
Aroclor - 1232								
Aroclor - 1242								
Aroclor - 1248								
Aroclor - 1254								
Aroclor - 1260								

* SEE EXHIBIT E, PART 7

** CONF. = CONFIRMATION (<20% DIFFERENCE)
QUANT. = QUANTITATION (<25% DIFFERENCE)

FORM IX

1/85

4.0 Instructions For Labeling Chromatograms (continued)

- Individual Standard Mix A or B (EPA-A and EPA-B)
- All multiresponse pesticides/PCB's
- All quantitation standards (includes DEDT series if run)
- A copy of the computer reproduction covering the 100 fold range

4.2 (QA) All chromatograms are required to have the following:

4.2.1 Standards:

- Labels for all standard peaks for all individual compounds either directly out from the peak or on the printout of retention times if retention times are printed over the peak.
- Label the chromatogram for multicomponent standards (i.e.: Aroclor 1242, Toxaphene, Chlordane).
- List concentration injected for each standard. (Above peak or in report printout.)
- A printout of retention times and corresponding peak areas must accompany each chromatogram.
- Date and time of injection
- GC column identification
- GC instrument identification
- Each case number of all the samples run with the set of standards for the day. If samples from two different cases are run, then both case numbers should be written on each standard chromatogram. (This labeling is for filing purposes.)

4.2.2 Samples:

Copies of pesticide chromatograms. All chromatograms must be labeled with the following information:

- Sample I.D. (SMO sample number from Traffic Report) including case number*.

*See explanation of Reagent Blank Summary, Form IV, found in Section 9.3 to find out how to label each reagent blank for different sets of circumstances.

4.0 Instructions for Labeling Chromatograms (continued)

- Volume injected (µl)
- Date and time of injection
- GC column identification
- GC instrument identification
- Positive identification must be labeled with the names of compounds, either directly out from the peak or on a printout of retention times if retention times are printed over the peak.

4.3 Copies of pesticide chromatograms from second GC column confirmation. Chromatograms to be labeled as in above instructions.

Header information common to most forms:

Contract Number : EPA 68-01-7025
Laboratory Name : ITAS-Knoxville
GC Column : Either 1.5% SP2250/1.95% SP2401 or 3% OV-1
GC Instrument ID: V-3740A or V-3740B or T-565-1

5.0 Interpretation of Chromatograms

5.1 The computer or integrator does not always label peaks correctly. With a list of retention time windows for each compound in the EPA-A and EPA-B standards as well as windows for all the other pesticides and PCB's run as standards, take the chromatograms and check them. Compare each peak's retention time with the list of windows. If it does not match a window, the computer/integrator should have marked it unknown in the report. If it is incorrectly labeled with a pesticide, simply draw a line through the identification. If a peak meets the window and is mislabeled or mislabeled as unknown, draw a line through the label and write the correct identification beside the label in the report. The computer/integrator will not identify any multiple peak compounds. As each chromatogram is evaluated, be sure to check for the multi-peak compound patterns. There may be combinations of compounds involved so remember to check for them all.

5.0 Interpretation of Chromatograms (continued)

5.2 Raw Data Sheet

5.2.1 After correctly labeling each peak in the report, fill out a column for that sample at that dilution on a raw data sheet for that GC column. An example of a raw data sheet for the mixed column follows. Label each page of raw data with the instrument ID, case number, and date of analysis. For example, this raw data sheet is filled out with V-3740A for the instrument ID; 5/25-26/85 for the date of analysis; and Case 4000 for the case number. If more than one case of samples is run on one day's chromatograms, fill out separate raw data sheets for each case.

5.2.2 In the blank top part of the first column, fill in the sample's SMO number, the ITAS sample number, and whether the sample is a water or low level or medium level soil. Fill in the date extracted (information found on prep sheet), the date analyzed, the run number (the file number from PE 7500 or LCI-100) and the dilution factor. If run at original, the dilution factor is 1.

5.2.3 Each compound listed on the raw data sheet has two lines. If a peak meets the window for a compound, fill in the top line for that compound with the peak area and the bottom line with the retention time of the peak. If there is a peak just barely outside the window for a compound, fill in the information for it and enclose the retention time in parentheses, indicating the retention time is outside the window.

5.2.4 Toxaphene and chlordane must be calculated differently so only a retention time should be recorded for them. This is also true of all the Aroclors except 1221.

5.2.5 Always leave a blank column after each column filled with peak areas and RT's for a sample. The blank column will be used to write in the calculated amounts of compounds found, whether or not the compound is confirmed (C = confirmed and NC = nonconfirmed) and if it is below the contract required detection limit (CRDL). See example of raw data sheet which follows.

5.2.6 Each chromatogram should be checked to see if it is labeled with (1) SMO case number, (2) SMO sample number, (3) injection volume, (4) instrument ID.

5.0 Interpretation of Chromatograms (continued)

5.2.7 After all notations and corrections have been made on sample chromatograms and all standards have been labeled, each chromatogram is to be initialed and dated by the person doing the interpretive and quantitative work. Any changes made after this must also be initialed and dated.

5.3 Copying Chromatograms

5.3.1 The chromatograms are then ready to be copied and reduced. The Canon copier does the best work. Use the top tray - letter size paper and reduce by 75%. Each chromatogram gets copied from the beginning as far forward as can be copied and from the end as far back to the front as can be copied so that there will be overlap.

5.3.2 The copies should be sorted into:

1. Evaluation standards: A, B, C, B, B, etc. in chronological order
2. EPA standards: A, B, A, B, etc. in chronological order
3. All other standards in chronological order
4. Samples: By sample and then in chronological order

The copies, which are then set aside, will go in the CLP data package.

5.4 Two different orders of events may be followed once the raw data form for the mixed column is completed. If the required confirmations have been run, then the QA (Quantitator-Analyst) can do the confirmation work before the calculations are done. This eliminates all the calculations for peaks which are not confirmed.

If on the other hand, the confirmation chromatograms have not been run, the QA may do the calculations from the mixed column. Using the Contract Required Detection Limits (CRDL), the QA will eliminate the peaks below the CRDL, thus eliminating the need for confirmation of those peaks.

6.0 Calculations

6.1 Single Peak Compounds

There are four calculation forms: two for each column for each matrix (soil and water). Calculations should be done on the mixed column

6.0 Calculations (continued)

when possible. The only exceptions to this are toxaphene, weathered chlordane calculated as alpha and gamma chlordanes, and those compounds whose % Difference on the Form IX - mixed column was greater than 15% but less than 20%.

6.1.1 Computer Generated Response Factors

Response factors will be printed in the GC report for EPA-A and EPA-B if the methods are updated as stated previously. D-BHC may be mislabeled as heptachlor. If so, calculate its response factor as described below. Use EPA-B for the dibutylchlorendate response factor.

6.1.2 Hand Calculation of Response Factors

Using a water calculation sheet for the GC column the chromatograms were run on, write in the concentration for each standard in the same column with the compound name and 0.1 for the dibutylchlorendate concentration. A list of concentrations for EPA-A and EPA-B follows. Use the list corresponding to the standards that were run. Write "response factors" as the sample number. Fill in the header information.

From the chromatograms of the first run of the day for EPA-A and EPA-B, fill in the peak areas for each compound. Use the peak area from EPA-B for dibutylchlorendate. Divide the concentration by the peak area for each compound to get its response factor.

6.1.3 Filling Out Calculation Forms

Using a blank calculation form for either water or sediment or both, depending on what sample matrices were run, fill in the header information leaving the sample number and SMO number blank. Copy the response factors for EPA-A, EPA-B, and dibutylchlorendate from either the updated reports from the chromatogram (6.1.1 above) or from the hand calculations (6.1.2 above). Make as many copies of this water and/or sediment form(s) as there are chromatograms of that matrix. Using the raw data sheet, fill in the sample number and SMO number for each sample - one injection per calculation sheet. Be sure to put water samples on water calculation sheets and soil samples on soil calculation sheets. Fill in the peak areas of compounds found and the dilution factor for that injection.

6.0 Calculations (continued)

6.1.4 Water Calculations

From the prep sheet, fill in the final volume (of extract, ~10 ml) on the α-BHC line. Next fill in the volume of sample extracted (~ 1000 ml) on the same line. The dilution factor should already be filled in. Draw arrows down the columns to the bottom line. See example following on Page . The water calculation is:

$$ppb = \frac{\text{peak area} \times \text{resp. factor} \times \text{final vol (ml)} \times \text{diln factor} \times 1000 \text{ ppb/ppm}}{\text{sample volume (ml)}}$$

6.1.5 Sediment Calculations

From the prep sheet, fill in the weight extracted, the final volume (of extract) and the dryness factor. Write in after the SMO number medium or a low prep. Draw lines with arrows in columns like the example calculation sheet on Page . The sediment calculation is:

$$ppb = \frac{\text{peak area} \times \text{resp. factor} \times \text{final vol (ml)} \times \text{diln factor} \times C}{\text{sample weight (g)} \times \text{dryness factor}}$$

where C = 10000 for medium level prep or
C = 20000 for low level prep

6.1.6 Checking CRDL

A copy of the CRDL (Contract Required Detection Limits) follows. Compare the calculated amount of each component to the CRDL. If the sample is a soil, the CRDL must be corrected for the dryness factor. To correct, divide the CRDL by the dryness factor. If the calculated amount is less than the CRDL, draw a line through the amount and write <CRDL beside it.

6.1.7 Filling in Raw Data Sheet

Transfer the calculated amounts from the calculation sheet to the raw data sheet. In the blank column write the amount for each compound next to the peak area of raw data sheet. If the amount is less than the CRDL, draw a line through it and write <CRDL below it on the sample line as the retention time.

6.0 Calculations (continued)

6.1.8 Filing Calculation Pages

The calculation pages are filed in the CLP package behind the raw data sheets which are behind the sample chromatograms.

6.2 Multicomponent Mixtures

For multicomponent mixtures (chlordane, toxaphene and PCB's) match retention times of peaks in the standards with peaks in the sample. Quantitate every identifiable peak (> 50% of the total area must be used) unless interference with individual peaks persist after cleanup. Add peak height or peak area of each identified peak in the chromatogram. Calculate as total response in the sample versus total response in the standard. An example calculation sheet for multicomponent mixtures follows.

6.2.1 Quantitation of Technical Chlordane

Weathering and/or different formulations of chlordane may modify the technical chlordane pattern shown in Figure . If the chlordane pattern in a sample is similar to Figure , use a technical chlordane standard for quantitation. If the pattern is different but gamma and alpha chlordane are present, use gamma and alpha chlordane standards for calculation, total the results, report under technical chlordane but footnote the data as calculated using gamma and alpha chlordane.

When the chlordane in the sample is a good match to the standard on the mixed column, calculate using as many of the five tallest peaks in the standard that are matched respectively in the sample. Add the peak areas of the five tallest peaks of the chlordane standard together. Divide the concentration of the standard by the total peak area to get the response factor (rf). On the sample calculation sheet, write the rf in the correct column on the line labeled technical chlordane. Add the peak areas of the five peaks in the sample chromatogram corresponding to the standards five tallest peaks in the standard. Enter this number in the column for peak area on the sample calculation sheet. Proceed with the calculation as described under single-peak calculation.

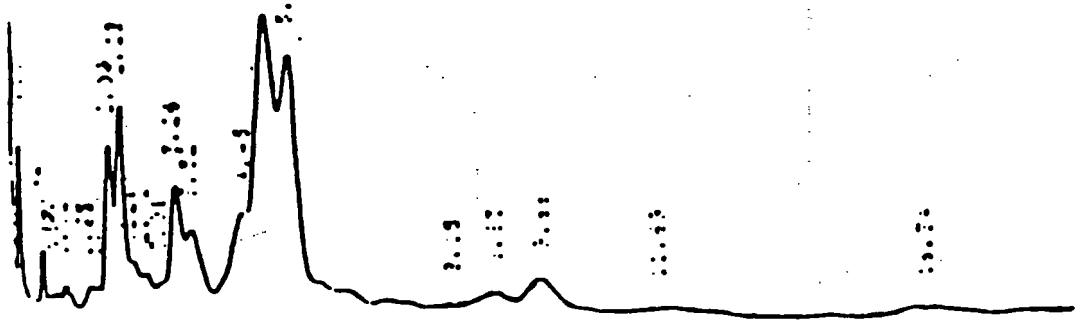


Figure 3. Gas chromatogram of technical chlordane. See Table 7 for conditions. (1.5% OV-17/1.95% OV-210)

6.0 Calculations (continued)

When the pattern of chlordane in the sample is altered from the standard's pattern on the mixed column, α and γ chlordanes are calculated on the OV-1 column. The concentration of α and γ chlordane in the technical chlordane standard are given on the list of standard concentrations and retention time windows at the end of the section (or use the most current list). Gamma (γ) chlordane is the first and alpha (α) is the second of the two tallest peaks in the technical chlordane stand. Calculate a response factor and then the quantity of α and γ chlordanes present using the instructions given under single-peak calculations.

6.2.2 Toxaphene

Calculate toxaphene on the column with the best separation of toxaphene from other peaks present in the chromatogram. Use peak height to calculate instead of peak area. Use as many peaks as are present in both standard and sample. Be sure to draw the baseline consistently in the same place in both sample and standard, using the peaks that are in common in both standard and sample and that also show no interference from anything else in the sample. Calculate a response factor for that sample by summing those peak heights and dividing the standard concentration by that sum. Sum the respective peak heights in the sample. Fill in the column on the sample calculation sheet labeled peak area with the sum of the peak heights of the sample's toxaphene and the rf calculated for that sample. Asterisk and footnote that toxaphene is calculated using peak height. Continue the calculation as explained under single-peak calculations.

6.2.3 Aroclors

Calculate aroclors on the column with the best separation from everything else present in the sample. Calculate like toxaphene, using as many peaks as match, in both sample and standard. Use peak areas. Do not use peaks with retention times matching those of pesticides. For example, Aroclors 1254 and 1260 have a peak that comes out at the same retention time of 4,4-DDT. Do not use this peak to calculate the aroclor if there is evidence of DDT in the sample. Use only the peaks that do not show interference when compared to the standard aroclor patterns. Again, sum the standard peak areas of the peaks common to both sample standard and divide the standard

6.0 Calculations (continued)

concentration by this sum. Sum the respective peak areas in the sample. Use the sample calculation sheet. Aroclors are not listed on it, so add the aroclor(s) found to the bottom of the sheet. Calculate as instructed under single peak calculations.

7.0 Confirmation

7.1 Check each sample chromatogram to see if peaks reported in the GC report meet the windows and check for any compounds identified on the other column. (Be sure to check all spikes for all spiking compounds.) All peaks should be correctly labeled in the data report following the chromatograms. Once all corrections are made, the QA will fill out a raw data sheet for the OV-1 column. Only retention times are needed for the peaks found to be within windows. The exceptions are toxaphene and any compound that did not meet quantitation specifications on Form IX for the mixed column.

7.2 Once each chromatogram is labeled and corrected, it should be initialed and dated by the QA. The chromatogram is then ready to be copied.

7.3 The raw data sheets from the mixed column and the OV-1 column are then compared. A 1/5 dilution of a sample on one column is compared to a 1/5 dilution on the other. Only peaks that are within the window on both columns are confirmed. Endrin will not meet the Endosulfan II/Endrin window on the OV-1 column because the coelution of the two compounds shifts the window later in time. However, for comparison purposes, the endrin in the closest Evaluation B can be used to check the retention time of a suspected endrin peak. If a peak confirms, a C should be written beside its retention time on both sets of raw data sheets. If a peak does not confirm, write NC beside the retention time.

7.4 If GC/MS confirmation is required (see contract for concentration level), inform GC/MS group in writing immediately.

8.0 Completion of the Sample Raw Data Package

Once all calculations and confirmations are complete, the copies of the chromatograms must be made.

Column 1.5% SP2250/ 1.9% SP2401 Temp. _____	BA202 Water B1312		BA203 Water B1313		BA204 Water B1314		BA204 MS B1315		BA204 MSD B1316	
Extracted	5-15		5-15		5-15		5-15		5-15	
Analyzed	5.21		5-21		5.21		5-21		5-21	
Run #	.010a		.011a		.012a		.013a		.014a	
Dilution Factor	1		1		1		1		1	
a-BHC	7023	.0077								
	1.92	<CRDL								
Lindane			30901	0.11			40678 (GS)	.19	40412 (GS)	.18
			(2.370)	NC			2.41	C	2.411	C
b-BHC										
Heptachlor							38960 (GS)	.17	38949 (GS)	.17
							2.92	C	2.922	C
-BHC	50129	0.081								
	3.12	C								
ldrin					6727	.032	40537	.12	40499	.11
					3.48	<CRDL	3.48	C	3.49	C
Hept Epoxide										
ndosulfan I										
DDE	408690	0.18								
	6.99	NC								
ieldrin							115068	.48	109003	.49
							7.50 (GS)	C	7.50 (GS)	C
n		0.12					106780	.37	100119	.40
	9.019	(NC)					9.02	NC	9.018	NC
DDD										
ndosulfan II	off scale	NC								
	10.32									
DDT							328614	.39	354109	.37
							12.17	C	12.18	C
ndrin Aldehyde										
do Sulfate										
BuChlorendate	1052667	2.6*	315800	0.78	331995	0.82	364385	0.90	344141	0.85
	20.71		20.71		20.72		20.70		20.72	
thoxychlor										
ndrin Ketone										
B 1221										
CB 1016										
B 1232										
CB 1242										
B 1248										
B 1254										
CB 1260										

raphene * Interference

(.5µl) T-565 6/26-27/85			(2.5µl) V-3740 B 6-24-85	
SP2250/1.95% SP2401 ak windows	1.5% SP2250/1.95% SP2401 Conc in Std	COMPOUND	3% OV-1 Conc. in std ·3/10	3% OV-1 Peak Windows
0.036	0.0124	alpha-BHC	0.00371	0.028
0.035	0.0212	beta-BHC	0.00636	0.029
0.046	0.0195	delta-BHC	0.00584	0.032
0.036	0.0118	gamma-BHC	0.00354	0.020
0.046	0.0102	Heptachlor	0.00305	0.046
0.046	0.0200	Aldrin	0.0060	0.031
0.096	0.0216	Heptachlor Epoxide	0.00647	0.045
0.114	0.0366	Endosulfan I	0.01098	0.047
0.131	0.0330	Dieldrin	0.00899	0.075
0.137	0.0446	4,4'-DDE	0.0134	0.131
0.182	0.0568	Endrin	0.03681	0.065
204	0.0659	Endosulfan II	(0.01704 + 0.01977)	
.200	0.0758	4,4'-DDD	0.05274	0.165
0.217	0.10	Endrin Aldehyde	(0.02274 + 0.03)	
0.251	0.0896	Endosulfan Sulfate	0.02688	0.200
0.217	0.0989	4,4'-DDT	0.02967	0.200
0.444	0.225	Methoxychlor	0.0675	0.167
0.453	0.10	Endrin Ketone	0.03	0.244
0.108		Tech. Chlordane		0.057
		alpha-Chlordane*	0.00595	0.095
		gamma-Chlordane*	0.00831	0.068
0.204		Toxaphene		0.204
0.040		Aroclor - 1016		0.024
0.013		Aroclor - 1221		0.012
0.037		Aroclor - 1232		0.015
043		Aroclor - 1242		0.025
0.051		Aroclor - 1248		0.009
0.131		Aroclor - 1254		0.030
0.478		Aroclor - 1260		0.142

Water Calculation Form

Case No. 4000 Contract Laboratory ITAS-Knoxville

Column 1.5% SP 2250/1.95% SP 2401 Date Analyzed 5-21-85

Sample # B-1313 SMO # BA203

Compound	Peak Area	Response Factor	Final Volume	Dilution Factor	Sample Volume	Conc (ppb)
alpha-BHC		1.890×10^{-7}	10 ml	1	1000 ml	
beta-BHC		2.642×10^{-7}				
delta-BHC		1.858×10^{-7}				
gamma-BHC	30901	1.827×10^{-7}				0.11
Heptachlor		1.268×10^{-7}				
Aldrin		1.580×10^{-7}				
Heptachlor Epoxide		1.507×10^{-7}				
Endosulfan I		1.741×10^{-7}				
Dieldrin		1.785×10^{-7}				
4,4'-DDE		2.104×10^{-7}				
Endrin		2.370×10^{-7}				
Endosulfan II		1.739×10^{-7}				
4,4'-DDD		2.335×10^{-7}				
Endrin Aldehyde		2.513×10^{-7}				
Endosulfan Sulfate		3.016×10^{-7}				
4,4'-DDT	–	4.006×10^{-7}				
Methoxychlor		4.238×10^{-7}				
Endrin Ketone		1.884×10^{-7}				
Tech. Chlordane						
alpha-Chlordane						
gamma-Chlordane						
Dibutylchlorendate	315800	2.516×10^{-7}				0.78

Sediment Calculation Form

Case No. 4000 Contract Laboratory ITAS-Knoxville

Column 1.5% SP2250/1.95% SP2401 Date Analyzed _____

Sample # B-1317 SMO # BA 205 Low

Compound	Peak Area	Response Factor	Final Volume	Dilution Factor	Sample Weight	Dryness Factor	Conc(ppb)
alpha-BHC		1.890×10^{-7}	1.0 ml	1	30.43	.5909	
beta-BHC		2.642×10^{-7}					
delta-BHC		1.898×10^{-7}					
gamma-BHC		1.827×10^{-7}					
Heptachlor	36804	1.268×10^{-7}					~~5.1~~ <CRDL
Aldrin		1.580×10^{-7}					
Heptachlor Epoxide		1.507×10^{-7}					
Endosulfan I	14940	1.741×10^{-7}					~~2.9~~ <CRDL
Dieldrin	67711	1.785×10^{-7}					~~13.~~ <CRDL
4,4'-DDE		2.104×10^{-7}					
Endrin		2.370×10^{-7}					
Endosulfan II		1.739×10^{-7}					
4,4'-DDD		2.335×10^{-7}					
Endrin Aldehyde		2.513×10^{-7}					
Endosulfan Sulfate		3.016×10^{-7}					
4,4'-DDT		4.006×10^{-7}					
Methoxychlor		4.238×10^{-7}					
Endrin Ketone		1.884×10^{-7}					
Tech. Chlordane							
pha-Chlordane							
gamma-Chlordane							
Dibutylchlorendate	385103	2.516×10^{-7}	↓	↓	↓	↓	110.

CRDL

Pesticides	CAS Number	Detection Limits* Low Water[e] ug/L	Low Soil/Sediment[f] ug/Kg	
			Med H_2O	Med Soil
104. alpha-BHC	319-84-6	0.05	5.	8.0	120.
105. beta-BHC	319-85-7	0.05	5.	8.0	120.
106. delta-BHC	319-86-8	0.05	5.	8.0	120.
107. gamma-BHC (Lindane)	58-89-9	0.05	5.	8.0	120.
108. Heptachlor	76-44-8	0.05	5.	8.0	120.
109. Aldrin	309-00-2	0.05	5.	8.0	120.
110. Heptachlor Epoxide	1024-57-3	0.05	5.	8.0	120.
111. Endosulfan I	959-98-8	0.05	5.	8.0	120.
112. Dieldrin	60-57-1	0.10	10.	16.0	240.
113. 4,4'-DDE	72-55-9	0.10	10.	16.0	240.
114. Endrin	72-20-8	0.10	10.	16.0	240.
115. Endosulfan II	33213-65-9	0.10	10.	16.0	240.
116. 4,4'-DDD	72-54-8	0.10	10.	16.0	240.
117. Endrin Aldehyde	7421-93-4	0.10	10.	16.0	240.
118. Endosulfan Sulfate	1031-07-8	0.10	10.	16.0	240.
119. 4,4'-DDT	50-29-3	0.10	10.	16.0	240.
120. Endrin Ketone	53494-70-5	0.10	10.	16.0	240.
121. Methoxychlor	72-43-5	0.5	50.	80.0	1200.
122. Chlordane	57-74-9	0.5	50.	80.0	1200.
123. Toxaphene	8001-35-2	1.0	100.	160.0	2400.
124. AROCLOR-1016	12674-11-2	0.5	50.	80.0	1200.
125. AROCLOR-1221	11104-28-2	0.5	50.	80.0	1200.
126. AROCLOR-1232	11141-16-5	0.5	50.	80.0	1200.
127. AROCLOR-1242	53469-21-9	0.5	50.	80.0	1200.
128. AROCLOR-1248	12672-29-6	0.5	50.	80.0	1200.
129. AROCLOR-1254	11097-69-1	1.0	100.	160.0	2400.
130. AROCLOR-1260	11096-82-5	1.0	100.	160.0	2400.

[e]Medium Water Contract Required Detection Limits (CRDL) for Pesticide HSL Compounds are 100 times the individual Low Water CRDL.

[f]Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Pesticide HSL compounds are 15 times the individual Low Soil/Sediment CRDL.

*Detection limits listed for soil/sediment are based on wet weight. The detection limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, as required by the contract, will be higher.

** Specific detection limits are highly matrix dependent. The detection limits listed herein are provided for guidance and may not always be achievable.

Case #: _____

Laboratory #: _____

Date Analyzed: _____

MULTI-PEAK COMPOUND CALCULATION SHEET

Compound:		Column:	
Standard:		Sample	
Concentration:			
RT	(circle one) Peak Area or Peak Height	RT	(circle one) Peak Area or Peak Height
	Total Peak Area Used:		Total Peak Area
	rf =		

8.0 Completion of the Sample Raw Data Package (continued)

8.1 Use the Canon copier if it is available. Copy from the start of the chromatogram as far toward the end as possible and a second time from the end back toward the beginning as far as possible. Reduce by 75% using the top "letter" sized paper tray.

8.2 Once each roll of chromatograms is copied, check each page to make sure all information is legible. Sort the copies into four stacks. Evaluation Standards go in the first stack with Evaluations A, B, C first and then each succeeding Evaluation B in chronological order. The EPA-A and EPA-B standards go in the second stack again in chronological order. The next stack is all the other standard chromatograms, again in chronological order. The last stack is the samples, again in chronological order.

8.3 After all the rolls of chromatograms are copied and sorted, separate the copies into two groups - one for each column. Each group should be arranged counter-chronologically with the most recent set of chromatograms on top and the oldest on bottom.

8.3.1 The most recent set of confirmation chromatograms is then sorted into the following piles: (1) Evaluation Standards, (2) EPA-A and EPA-B, (3) all other standards and then a pile for each sample run. Then the next most recent set of chromatograms is sorted onto the same piles and so on until all the confirmation chromatograms have been sorted. Next, the most recent quantitation set of chromatograms is sorted onto the piles and so on until all the chromatograms have been sorted.

8.3.2 When the sorting is complete, there should be a stack for each sample, for each matrix spike, for each matrix spike duplicate, for each reagent blank and a stack for the Evaluation Standards, the EPA-mix standards, and a stack for all the other standards. Each stack should be in the order by column of quantitation first and confirmation last and chronologically ordered inside those two categories.

8.3.3 Going back to the raw data sheets, each page should have as many copies as there are different samples listed on it. If a page has a sample with two different dilutions on it, just one copy is needed for that sample.

8.3.4 Each stack of sample chromatograms should have a raw data sheet for each separate chromatogram, both quantitative and confirmative. The calculation sheets corresponding to the raw data

8.0 Completion of the Sample Raw Data Package (continued)

sheets should be put behind the raw data sheet. Any other calculation sheets such as list of retention times and peak areas or heights for calculation of multi-component compounds should also be included. Each sample should have the minimum of one chromatogram, one raw data sheet and one calculation sheet. Even if nothing is reported in the sample, the surrogate recovery must be calculated. Graphs for DDE, DDD, and DDT when linearity is greater than 10% RSD are also included.

8.3.5 The stacks of standard calculations are combined with Evaluation Standards on top, next EPA mixed standards, and the stack of all the other standards on bottom.

8.3.6 If more than one case was run on the same day, make as many copies of Forms VIII and IX for that day as were cases run. File the originals with the first case run in the purge file. Purge file is described later.

8.3.7 Then fill in the case number on Forms VIII and IX. Put the copies for other cases aside until they are needed. Make sure the case number is written on each form that requires it.

8.3.8 Fill in the SMO sample number for each injection made on the bottom of Form VIII. This information is found on the sample receipt log. A copy is kept on Nancy's desk.

All the Form VIII's and IX's are then sorted into the same relative order as the chromatograms: by column and then chronologically for each column. These are added to the stack of standard chromatograms. This is all of the standards package except for Form X. Form X is the last to be filled out so the standards package is set aside for now. The forms for the linearity checks of the DEDT standards are filed behind Form VIII.

9.0 Filling Out the Organic Analysis Data Sheet (OADS), p3

9.1

SMO No. _____

Laboratory Name - ITAS-Knoxville
Case No. _____

Concentration Low Medium ← Water is always low; soil is either low or medium. See prep sheet.

Date extracted/prepared _____ ← Date on prep sheet

Date analyzed _____ ← Every date that this sample ran. See copies of chromatograms in stack for this sample.

Conc./Dil. Factor _____ ← Circle dil. factor and write in dil. factors used, for original use 1: µg/l or µg/kg water is µg/l
circle units soil is µg/kg

9.2 Fill in each calculated amount for each compound detected that is over the CRDL. Use two significant figures only. If a compound was not detected, write the detection limit and U (for example: 0.05U or 8.U).

V_s = volume of water extracted - should be 1000 ml or close to it

W_s = weight of sample extracted - ~30g or ~1g

V_t = volume of total extract - 10000 µl for water and medium soils; 20000 µl for low soils

V_i = injection volume - 4 µl for V-3740A, 2.5 µl for V-3740B, 5 µl for T-565

See example following.

Laboratory Name ITAS-Knoxville

Case No 4000

Sample Number BA-202

Organics Analysis Data Sheet (Page 3)

Pesticide/PCBs

Concentration: (Low) Medium (Circle One)

Date Extracted/Prepared: 5-15-85

Date Analyzed 5/20,21,29/85 6/1,2,5/85

Conc/(Dil Factor): 1, Vs

CAS Number		(ug/l) or ug/Kg (Circle One)
319-84-6	Alpha-BHC	0.054
319-85-7	Beta-BHC	0.054
319-86-8	Delta-BHC	0.081
58-89-9	Gamma-BHC (Lindane)	0.054
76-44-8	Heptachlor	0.054
309-00-2	Aldrin	0.054
1024-57-3	Heptachlor Epoxide	0.054
959-98-8	Endosulfan I	0.054
80-57-1	Dieldrin	0.14
72-55-9	4,4'-DDE	0.14
72-20-8	Endrin	0.12
33213-65-9	Endosulfan II	0.14
72-54-8	4,4'-DDD	0.14
7421-93-4	Endrin Aldehyde	0.14
1031-07-8	Endosulfan Sulfate	0.14
50-29-3	4,4'-DDT	0.14
72-43-5	Methoxychlor	0.54
53494-70-5	Endrin Ketone	0.14
57-74-9	Chlordane	0.54
8001-35-2	Toxaphene	1.4
12674-11-2	Aroclor-1016	0.54
11104-28-2	Aroclor-1221	0.54
11141-16-5	Aroclor-1232	0.54
53469-21-9	Aroclor-1242	0.54
12672-29-6	Aroclor-1248	0.54
11097-69-1	Aroclor-1254	1.4
11096-82-5	Aroclor-1260	1.4

V_i = Volume of extract injected (ul)

V_s = Volume of water extracted (ml)

W_s = Weight of sample extracted (g)

V_t = Volume of total extract (ul)

V_s 1000 ml or W_s _____ V_t 10000 ul V_i 4 ul, 2.5 ul

9.0 Filling out the Organic Analysis Data Sheet (OADS), p3 (continued)

9.3 All of the reagent blanks are then given an OADS p3 form and filled out. If the case contains only water samples, there is only one reagent blank and it is given the label MB1-XXXX, where XXXX is the case number.

9.3.1 If the case contains only sediments of one level, there is only one reagent blank and it is labeled MB1-XXXX, where XXXX is the case number. If there are sediments only in the case but two levels of prep, the low level sediment reagent blank is MB1-XXXX and the medium level is MB2-XXXX.

9.3.2 If the case contains both water and sediments, the matrix with the lowest SMO number sample number in it has the MB1-XXXX reagent blank number. The other matrix gets MB2-XXXX. If there are two levels of sediments, the lower level sediment gets the lower blank number (MB1 or MB2 depending on whether or not the water or the soil matrix has the sample with the lowest SMO number) and the medium level blank gets the MB3-XXXX number.

9.4 The header information is filled out according to the general OADS instructions. If anything was detected above CRDL, report it on the OADS and also on the Reagent Blank Summary, Form IV.

A copy of Form IV follows. It has been filled out for Case 4000 which had two matrices and sediments at two levels. Nothing was detected in any of the blanks.

9.5 On calculating recoveries for reagent blanks for sediments, the weights of 1.00g for medium level and 30.00g for low level are assumed for wt of sample extracted. A dryness factor of 1.000 is also assumed. If anything is found at a level at or above the detection limit, then if that compound is found in a sample, the level is reported with a "B" qualifier. See OADS page 1 for "B" footnote.

9.6 Anything reported as detected must also be listed on Form X.

9.7 If any soils are analyzed, the list of CRDL's must be corrected for the dryness factor. Divide the CRDL by the dryness factor and round to one significant figure if it is below ten and to two significant figures if it is above ten. These detection limits apply to this sample only.

REAGENT B. NK SUMMARY

Case No. 4000 Contractor ITAS-Knoxville Contract No. EPA 68-01-7025

FILE ID	DATE OF ANALYSIS	FRACTION	MATRIX	CONC. LEVEL	INST. ID	CAS NUMBER	COMPOUND (HSL, TIC OR UNKNOWN)	CONC.	UNITS	CRDL
MB1-4000	6-1,2,12,15-85	Pest	Water	Low	V-3740A V-3740B		nothing detected			
MB2-4000	5-29-31-85 6-1,3&9-85	↓	Soil	Low	V-3740A V-3740B		↓			
MB3-4000	6-1,3,8-85	↓	Soil	Med	V-3740A V-3740B		↓			

Comments:

FORM IV

9.0 Filling Out the Organic Analysis Data Sheet (OADS), p3 (continued)

9.8 Each sample gets an OADS p3. Be sure to use the corrected CRDL for soils. Do all the samples before doing the matrix spikes/matrix spike duplicates.

9.9 All of the completed samples (the completed OADS p3 goes on top of the stack of chromatograms, raw data sheets and calculation sheets) are then put in increasing SMO number and clipped together. This is the samples package for pesticides/PCB's.

10.0 Reporting the Spikes

On the OADS p3, an S should be put in the right-hand side of the column for reporting sample results next to the compounds in the spiking solution: lindane, heptachlor, aldrin, dieldrin, endrin and 4,4'-DDT. The OADS p3 is then footnoted with: S = spiked compound.

10.1 Water MS/MSD

Report all compounds found in the sample including the spike compounds on the OADS p3. Fill in Form X for everything reported. Then fill in Form III.

10.1.1 For water samples, the concentration of spike added is calculated as follows:

Q_A = Quantity Added Q_D = Quantity determined

Q_A for lindane, heptachlor and aldrin:

$$\frac{X^* \ \mu l \times (1 \ ml/1000 \ \mu l) \times 0.2 \ \mu g/1. \ ml}{\text{sample volume extracted (l)}} = \mu g/l$$

* where X is the # of µl of spiking solution added to the sample

Q_A for dieldrin, endrin, 4,4'-DDT:

$$\frac{X^* \ \mu l \ (1 \ ml/1000 \ \mu l) \times 0.5 \ \mu g/1 \ ml}{\text{sample volume extracted (l)}} = \mu g/l$$

10.0 Reporting the Spikes (continued)

10.1.2 Fill in the Q_A for the spiked compounds on Form III. Fill in the SMO number for the sample spiked. Fill in the Case number. Now, refer to the OADS p3 for the original sample which was also used for the spike. Report under sample result any spike compound found in that sample. If none were found, write "0". Now fill in the concentrations reported on the OADS p3 for the matrix spike (MS) and the matrix spike duplicate (MSD). Using the formulae below, calculate percent recovery (% Rec) and relative percent difference (RPD). Fill these in. Asterisk and footnote any results outside the limits on the right-hand side of the page.

$$\text{Matrix spike percent recovery} = \frac{SSR - SR}{SA} \times 100$$

where SSR = spike sample results
SR = sample result
SA = spike added from spiking mix

$$\text{Relative percent difference} = RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

where RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (duplicate)

10.1.3 If the sample has high concentrations of pesticides in it so that the spiked compounds are diluted out, fill in DL in the spaces to report conc MS and conc MSD and footnote.

10.1.4 Fill in the bottom part of the form. For each MS/MSD there are 12 recoveries to report. On the line that says "Pest _____ out of 12; outside QC limits," under recovery, fill in the number of recoveries missed out of the total of 12. On the line, "RPD: Pest _____ out of 6; outside QC limits," write in the number of RPD's outside the limits.

10.2 Soil MS/MSD (both low and medium level preps)

Report all compounds confirmed and detected above CRDL on the OADS p3 for both samples. Fill in Form X for both samples. Then fill in Form III. Fill in the case number and whether the prep is low or medium. Fill in the sample number under Pest SMO Sample No.

10.0 Reporting the Spikes (continued)

10.2.1 Calculate the concentration of spike added (µg/kg) using the formulae given below:

Quantity added: Q_A X µl = µl of spiking soln
Low level prep - 30g, Medium level prep - 1g

For lindane, heptachlor, and aldrin:

$$Q_A = \frac{X\ \mu l \times (1\ ml/1000\ \mu l)\ (2\ \mu g/ml)\ (1000\ g/kg)}{\text{sample wt (- 30g or - 1g) x dryness factor}} = \mu g/kg$$

For dieldrin, endrin, and DDT:

$$Q_A = \frac{X\ \mu l\ (1\ ml/1000\ \mu l)\ (5\ \mu g/ml)\ (1000\ g/kg)}{\text{sample wt (- 30g or - 1g) (dryness factor)}} = \mu g/kg$$

10.2.2 Fill in the Q_A for the spiked compounds. Refer back to the OADS p3 for the sample levels of spiked compounds. If the level is below CRDL, write 0 in the box labeled sample result.

Now fill in the amounts for each compound reported on the OADS p3 for the MS and the MSD. Calculate % Rec and RPD using the following formulae.

$$\text{Matrix spike percent recovery} = \frac{SSR - SR}{SA} \times 100$$

where SSR = spike sample results
SR = sample result
SA = spike added from spiking mix

$$\text{Relative percent difference} = RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} \times 100$$

where RPD = relative percent difference
D_1 = first sample value
D_2 = second sample value (duplicate)

ATER MATRIX SPIKE/MAT SPIKE DUPLICATE RECOVERY

Case No. 4000 Contractor ITAS-Knoxville Contract No. EPA 68-01-7025

FRACTION	COMPOUND	CONC. SPIKE ADDED (ug/L)	SAMPLE RESULT	CONC. MS	% REC	CONC. MSD	% REC	RPD	QC LIMITS * RPD	QC LIMITS * RECOVERY
VOA SMO SAMPLE NO.	1,1-Dichloroethene									
	Trichloroethene								14	61-145
	Chlorobenzene								14	71-120
	Toluene								13	75-130
_____	Benzene								13	76-125
B/N SMO SAMPLE NO.	1,2,4-Trichlorobenzene								11	78-127
	Acenaphthene								28	39-98
	2,4 Dinitrotoluene								31	46-118
	Di-n-Butylphthalate								38	24-96
	Pyrene								40	11-117
	N-Nitroso-Di-n-Propylamine								31	26-127
_____	1,4-Dichlorobenzene								38	41-116
ACID SMO SAMPLE NO.	Pentachlorophenol								28	36-97
	Phenol								50	9-103
	2-Chlorophenol								42	12-89
	4-Chloro-3-Methylphenol								40	27-123
_____	4-Nitrophenol								42	23-97
PEST SMO SAMPLE NO.	Lindane	.2	0	.15	75%	.14	70%	7%	50	10-80
	Heptachlor	.2	0	.14	70%	.15	75%	7%	15	56-123
	Aldrin	.2	0	.12	60%	.11	55%	9%	20	40-131
	Dieldrin	.5	0	.36	72%	.39	78%	8%	22	40-120
	Endrin	.5	0	.37	74%	.40	80%	8%	18	52-126
BA204	4,4'-DDT	.5	0	.34	68%	.37	74%	8%	21	56-121
									27	38-127

* ASTERISKED VALUES ARE OUTSIDE QC LIMITS.

RPD: VOAs ____ out of ____ : outside QC limits
B/N ____ out of ____ : outside QC limits
ACID ____ out of ____ : outside QC limits
PEST 0 out of 6 : outside QC limits

RECOVERY: VOAs ____ out of ____ : outside QC limits
B/N ____ out of ____ : outside QC limits
ACID ____ out of ____ : outside QC limits
PEST 0 out of 12 : outside QC limits

Comments: _____

SOIL MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Case No. 4000 Contractor ITAS-Knoxville Contract No. EPA 68-01-7025

Low Level X Medium Level _____

FRACTION	COMPOUND	CONC. SPIKE ADDED (ug/Kg)	SAMPLE RESULT	CONC. MS	% REC	CONC. MSD	% REC	RPD	QC LIMITS * RPD	QC LIMITS * RECOVERY
VOA SMO SAMPLE NO. _____	1,1-Dichloroethene								22	59-172
	Trichloroethene								24	62-137
	Chlorobenzene								21	60-133
	Toluene								21	59-139
	Benzene								21	66-142
B/N SMO SAMPLE NO. _____	1,2,4-Trichlorobenzene								23	38-107
	Acenaphthene								19	31-137
	2,4-Dinitrotoluene								47	28-89
	Di-n-Butylphthalate								47	29-135
	Pyrene								36	35-142
	N-Nitrosodi-n-Propylamine								38	41-126
	1,4-Dichlorobenzene								27	28-104
ACID SMO SAMPLE NO. _____	Pentachlorophenol								47	17-109
	Phenol								35	26-90
	2-Chlorophenol								50	25-102
	4-Chloro-3-Methylphenol								33	26-103
	4-Nitrophenol								50	11-114
PEST SMO SAMPLE NO. BA210	Lindane	27.	0	25.	93%	26.	96%	4%	50	46-127
	Heptachlor	27.	0	23.	85%	24.	89%	4%	31	35-130
	Aldrin	27.	0	23.	85%	22.	81%	4%	43	34-132
	Dieldrin	67.	0	60.	90%	58.	87%	3%	38	31-134
	Endrin	67.	0	55.	82%	50.	75%	10%	45	42-139
	4,4'-DDT	67.	0	49.	73%	45.	67%	9%	50	23-134

*ASTERISKED VALUES ARE OUTSIDE QC LIMITS.

RPD: VOAs ____ out of ____ : outside QC limits
B/N ____ out of ____ : outside QC limits
ACID ____ out of ____ : outside QC limits
PEST 0 out of 6 : outside QC limits

RECOVERY: VOAs ____ out of ____ : outside QC limits
B/N ____ out of ____ : outside QC limits
ACID ____ out of ____ : outside QC limits
PEST 0 out of 12 : outside QC limits

Comments: _____

10.0 Reporting the Spikes (continued)

10.2.3 If the spike compounds were diluted out due to high concentrations of pesticides in the sample matrix, write DL in for the conc MS and conc MSD values and footnote.

10.2.4 For the values for % Rec and RPD that are outside the QC limits given in the two far right-hand columns, asterisk the value and footnote why it is outside on the bottom of the form. Fill in the bottom of the form stating how many of the 6 RPD values were missed in the pesticide section and how many of 12 % Rec were missed for the pesticide section.

A copy of the soil MS/MSD recovery form follows.

11.0 Form X - Pesticide/PCB Identification

11.1 Form X is filled out for each compound reported in each sample. If nothing is detected, then that sample is not listed on Form X.

11.2 Form X is always filled out for every case. If nothing else is found, the six spiking compounds are reported in both the spike and the spike dupicate. These must also be reported on Form X.

11.3 For every compound reported, the retention time and the windows on both columns are filled in. GC/MS confirmation is usually not done because it requires a minimum of 10 ppm in the extract for most pesticides.

11.3.1 For the retention times, refer to the raw data sheets - the originals. For the windows, refer to the computer printouts for the day the sample was run that the number was reported.

11.4 Once Form X has been completely filled in after all the OADS p3 are complete, then Form X should be added to the standards package set aside previously. The order of the standards package is: Current Instrument Detection Limits; Evaluation Standards Form VIII - all of them; Pesticide/PCB Standards Summary Form IX; Form X; copies of Evaluation Standards; copies of EPA - mixed standards; copies of all other standards. The whole stack is clipped together and labeled Standards Package.

Pesticide, B Identification

Case No. 4000

Contract No. EPA 68-01-7025

Laboratory ITAS-Knoxville

SAMPLE ID	PRIMARY COLUMN	PESTICIDE/ PCB	RT OF TENTATIVE ID	RT WINDOW OF APPROPRIATE STANDARD	CONFIRMATION COLUMN	RT ON CONFIRMATORY COLUMN	RT WINDOW OF APPROPRIATE STANDARD	GC/MS CONFIRMED (Y or N)
BA202	1.5%@2230 1.5%@2401	D-BHC	3.12	3.064 - 3.156	3%OV-1	3.69	3.663-3.727	N
↓		Endrin	9.019	8.838 -9.202		8.198 *	8.62-8.68	
BA-204 MS		Lindane	2.41	2.376-2.444		2.154	2.143-2.165	
		Heptachlor	2.92	2.893-2.947		3.48	3.463-3.497	
		Aldrin	3.48	3.434-3.526		4.31	4.293-4.327	
		Dieldrin	7.50	7.386-7.614		7.64	7.61-7.67	
		Endrin	9.02	8.838-9.202		8.20 *	8.62-8.68	
		4,4'-DDT	12.17	11.943-12.397		12.50	12.30-12.70	
BA-204MSD		Lindane	2.411	2.376-2.444		2.156	2.143-2.165	
		Heptachlor	2.922	2.893-2.947		3.47	3.463-3.497	
		Aldrin	3.49	3.434-3.526		4.31	4.293-4.327	
		Dieldrin	7.50	7.386-7.614		7.63	7.61-7.67	
		Endrin	9.018	8.838-9.202		8.21 *	8.62-8.68	
		4,4'-DDT	12.18	11.943-12.397		12.52	12.30-12.70	
BA-210 MS		Lindane	2.43	2.376-2.444		2.155	2.143-2.165	
		Heptachlor	2.931	2.901-2.955		3.47	3.463-3.497	
		Aldrin	3.46	3.424-3.516		4.30	4.293-4.327	
		Dieldrin	7.51	7.390-7.618		7.62	7.61-7.67	
		Endrin	9.00	8.840-9.204		8.21 *	8.62-8.68	
		4,4'-DDT	12.15	11.940-12.394		12.34	12.30-12.70	
BA-210 MSD		Lindane	2.44	2.376-2.444		2.158	2.143-2.165	
		Heptachlor	2.925.	2.901-2.955		3.48	3.463-3.497	
		Aldrin	3.47	3.424-3.516		4.31	4.293-4.327	
		Dieldrin	7.52	7.390-7.618		7.62	7.61-7.67	
		Endrin	9.01	8.840-9.204		8.21 *	8.62-8.68	
↓	↓	4,4'-DDT	12.16	11.940-12.394	↓	12.40	12.30-12.70	↓

Form I

11.0 Form X - Pesticide/PCB Identification (continued)

11.5 Once all the OADS p3 have been filled in and attached to the corresponding stack of copies of chromatograms, raw data sheets and calculation sheets, the sample data package and the raw QC data package can be assembled.

11.6 The QC raw data package has all the matrix blanks in numerical order and then each pair of MS/MSD's in increasing SMO sample number. These are clipped together and labeled Raw QC Data Package.

11.7 The samples data package is simply all the sample packets arranged in increasing SMO sample number from front to back. These are clipped together and labeled Sample Data Package.

12.0 Surrogate % Recovery

12.1 Water Surrogate Recovery

The Quantity Added (Q_A) of surrogate (dibutylchlorendate) for water samples is calculated using the formula given below:

$$Q_A = \frac{X\ \mu l\ (1\ ml/1000\ \mu l)\ (1\ \mu g/ml)}{\text{sample volume (l)}} = \mu g/l$$

where X µl is the number of µl of surrogate spiking solution added to the sample.

12.1.1 Percent Recovery of the surrogate is calculated using the following formula:

Calculation for surrogate recovery

$$\text{Percent recovery} = \frac{Q_D}{Q_A} \times 100\%$$

where Q_D = quantity determined by analysis
Q_A = quantity added to sample

12.1.2 The percent recovery is filled in on Form II, Water Surrogate Percent Recovery Summary. The SMO sample number goes in the far left-hand column and the percent recovery goes in the far right-hand column. Fill in how many recoveries were outside of limits out of the total number of

12.0 Surrogate Percent Recovery (continued)

samples. Footnote any recoveries outside the limits given in the heading of the column. Also footnote if any were not calculated due to interference or being diluted out due to high concentration of pesticides or PCB's in the sample.

An example of Form II for water follows.

12.2 Soil Surrogate Recovery

12.2.1 The Quantity Added (Q_A) of surrogate (dibutylchlorendate) for soil samples is calculated using the following formula:

$$Q_A = \frac{X\ \mu l\ (1\ ml/1000\ \mu l)(20\ \mu g/ml)(1000\ g/kg)}{\text{sample wt (} \sim 30g \text{ or } \sim 1g) \times \text{dryness factor}} = \mu g/kg$$

where X µl = number of µl of surrogate spiking solution added to sample.

Q_A is different for each sample because the dryness factor is different in each sample.

12.2.2 Calculation for surrogate recovery

$$\text{Percent Recovery} = \frac{Q_D}{Q_A} \times 100\%$$

where Q_D = quantity determined by analysis
Q_A = quantity added to sample

12.2.3 Fill in Form II for sediments with the case number, the SMO sample numbers in the far left column and the percent recoveries in the far right column. Each level of sediment prepped gets a different Form II filled out. Asterisk and footnote any values outside the limits listed at the head of the column.

An example of low prep sediment surrogate recoveries reported on Form II follows.

WATER SURROGATE PERCENT RECOVERY SUMMARY

Case N 4000 Contract Laboratory ITAS-Knoxville Contract No. EPA 68-01-7025

	VOLATILE			SEMI-VOLATILE								PESTICIDE
SMO TRAFFIC NO.	TOLUENE-D8 (88-119)	BFB (83-121)	1,2 DICHLORO-ETHANE-D4 (77-120)	NITRO-BENZENE-D5 (41-120)	2-FLUORO-BIPHENYL (44-119)	TERPHENYL-D14 (33-128)			PHENOL-D5 (15-96)	2-FLUORO-PHENOL (23-107)	2,4,6 TRIBROMO PHENOL (20-105)	DIBUTYL-CHLORENDATE ** (48-138)
MBL-4000												
BA 202												81%
BA 203												Interference *
BA 204												78%
BA204MS												82%
BA204MSD												90%
												85%

* VALUES ARE OUTSIDE OF CONTRACT REQUIRED QC LIMITS

** ADVISORY LIMITS ONLY

Volatiles: _____ out of _____ ; outside of QC limits

Semi-Volatiles: _____ out of _____ ; outside of QC limits

Pesticides: 1 out of 6 ; outside of QC limits

Comments: * Interfering peak present on both columns. Dibutyl chlorendate present as a shoulder on OV-1 column but not able to be calculated.

SURROGATE PER[illegible]IT RECOVERY SUMMARY

Case No. 4000 Contract Laboratory ITAS-[illegible]ville Contract No. EPA 68-01-70[illegible]

Low X Medium _____

	VOLATILE				SEMI-VOLATILE							PESTICIDE
SMO TRAFFIC NO.	TOLUENE-D8 (50-160)	BFB (50-160)	1,2 DICHLOROETHANE-D6 (50-160)	NITROBENZENE-D5 (20-140)	2-FLUOROBIPHENYL (20-140)	TERPHENYL-D14 (20-150)			PHENOL-D5 (20-140)	2-FLUOROPHENOL (20-140)	2,4,6 TRIBROMOPHENOL (10-140)	DIBUTYLCHLORENDATE ** (20-150)
BA 205												82%
BA206												79%
BA207												83%
BA208												78%
BA209												62%
BA210												60%
BA210MS												55%
BA210MSD												81%
MB2 4000												58%

* VALUES ARE OUTSIDE OF CONTRACT REQUIRED QC LIMITS

** ADVISORY LIMITS ONLY

Volatiles: _____ out of _____ ; outside of QC limits

Semi-Volatiles: _____ out of _____ ; outside of QC limits

Pesticides: 0 out of 9 ; outside of QC limits

Comments: _____

13.0 Completion of Package

13.1 Once all forms have been filled out for blank, spike, and surrogate analyses, the forms are compiled in the following order:

A. Surrogate % Recovery Summary - Form II - all matrices and levels
B. MS/MSD Summary - Form III - all matrices and levels
C. Reagent Blank Summary - Form IV

These should be clipped together and labeled QC Summary.

13.2 The completed parts of the package are then organized in the following order:

1. QC Summary
2. Sample Data
3. Standards Data
4. Raw QC Data

13.3 Once organized, the package is put in an expandable file and given to analysis coordinator for spot checking.

13.4 After spot checking, the entire data package is given to the document control officer.

14.0 Purge File

14.1 Each roll of original chromatograms is folded up along the perforations. If more than one case of samples is run on a roll, then all the standards including the Evaluation B and mixed pesticides run intermittently among the samples are filed with the first case run. All the pieces of chromatogram for both cases are labeled as to which case the standards are filed with.

14.2 The originals of the computer program output sheets, the raw data sheets, and other calculation sheets for multi-peak compounds are also filed with the original chromatograms.

14.3 The original prep sheet and the GC project sheet are also filed in the purge file. The autosampler GC run log sheets are filed in the purge file. Once everything is filed in the purge file (use an expandable brown folder), give the purge file to the document control officer.

STANDARD OPERATING PROCEDURE

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: Data Review Procedure for CLP Pesticide Package				SOP NO: GC860529R2 DATE INITIATED: 05/29/86 REVISION NO: 2 DATE REVISED: 02/17/88 PAGE 1 of 4
PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Elizabeth G Tourney	Jack A Hall	3-10-88	Mary E Tyler	3-10-88

1.0 Scope and Application

This SOP outlines the items that are checked in the "Data Review" of a CLP pesticide package. This review is made on the completed data package before it is sent to the CLP Document Coordinator. This SOP covers those packages assembled manually.

The checks are made by a qualified person other than the analyst who prepared the package. A qualified person may be designated as the reviewer by the Technical Specialist, the Contract Coordinator, or the Laboratory Director.

0 Procedure

Check the following items. The order in which the checks are listed is the suggested review order.

General information common to all forms:

Lab Name: ITAS-Knoxville
Contract: 68-01-7468 (only for EPA CLP cases)
Lab Code: IT-STU
Case No: Use project code if not EPA CLP case
SAS No: Only for SAS EPA projects
SDG No: The lowest SMO sample ID or client ID in the case/project

2.1 Standards Package

2.1.1 Forms -

- Form VIII - pages 1 & 2, and Form IX's for each run sequence are present. Form X present if compounds are reported.
- % RSD $\leq$ 10%. If over, proper corrective action was taken.

2.0 Procedure (continued)

- If % RSD > 10% for DDT only, DDE, DDD, DDT linearity series was run. Graphs plotted if required.
- % Breakdown for DDT and Endrin ≤ 20%.
- Proper analytical sequence.
- % Difference for Dibutyl Chlorendate ≤ 2%.
- Any data not included in case has EPA SMO ID of ZZZZZ.
- Documentation concerning the analytical sequence as required in the narrative.
- Any corrections on forms are initialed and dated.
- Make needed corrections on forms, initial and date.
- Verify presence of Form IX for quantitation standards listed on Form VIII.
- % Difference for calibration factors ≤ 15% for quantitation and ≤ 20% for confirmation.
- DDT retention time window is reported for each 72-hour analytical sequence.
- Verify data system calculation of response factors.
- Check computer input for linearity and calibration factor % difference calculations.

2.1.2 Chromatograms

- Chromatograms labeled with Case No., sample ID, instrument, date and time of injection, column, and volume injected.
- Standard peaks labeled with peak ID and nanograms injected.
- Copies of AR1221 and AR1232 chromatograms included with the first sequence's chromatograms. Verify that these chromatograms are labeled with the correct case number or project code and that their run dates are within thirty days prior to sample analysis.

  AR1221 and AR1232 must be analyzed once each 30 days, and each primary run sequence for sample analysis must fall within the 30-day period of calibration for those aroclors.

2.0 Procedure (continued)

- Verify presence and chronological order of chromatograms for standards listed in the analytical sequence on each Form VIII. Verify that dates and times of injection on Form VIII, page 1 (EVALB injections), and page 2 agree with the date and time found on the chromatogram.
- Analyst has initialed and dated chromatogram at beginning of chromatogram and beside the chromatogram report.
- Initial and date any corrections or additions made by data reviewer.

2.2 QC Summary

- Forms II, III, and IV are present.
- No HSL compounds found $\geq$ CRDL in blanks.
- Surrogate % recoveries within suggested values. Verify results for any sample outside the window. (Check all calculations.)
- Spike recoveries and RPD inside windows. If not, verify results. (Check identifications and all calculations.)
- Forms filled out completely.
- Proper form used - water/soil.
- Any corrections are initialed and dated.

2.3 Sample Data and Raw QC Data

Check at least 20% of sample packages. Check all blank packages. A package consists of Form I, chromatograms, raw data sheet, calculation sheets/computer calculation sheets.

- Form I filled out completely and correctly. Check sample type, date received, date extracted, date analyzed, dilution, sample volume or weight used, extraction level, extraction type, GPC clean-up, pH, and SMO # or client ID.
- Dates, times, and dilutions on chromatograms match those listed on Form I.
- All raw data and calculation sheets are initialed and dated.
- Chromatograms are labeled with Case No., sample ID, instrument ID, column ID, date and time of injection, volume injected, and analyst's initials and date.

2.0 Procedure (continued)

- Chromatogram peaks - positive identifications are labeled with name of compound, either above the peak or on the data system report.
- Remove unnecessary chromatograms and make corrections to all forms involved.
- Verify identification of all peaks.
- Check for confirmation of positive ID's on primary column.
- Verify results on raw data sheet.
- Verify input into computer - computer calculations.
- Verify manual calculations.
- Initial and date any corrections made.
- Initial and date each chromatogram checked.
- Initial and date raw data and calculation sheets checked.
- Verify that results on Form I match results on calculation forms.
- Footnotes are present when needed.
- Reviewer accepts identification of compounds and method of calculation used.
- Form X filled out correctly with sample information.
- Only 2 dilutions submitted for each sample or QC sample.

2.4 Overall Package Check

- All forms completely filled out.
- Separate sections in order.
- Address any analytical problems or noncompliance of data in the case narrative. Explain any problems and document corrective action taken.

Data Review Procedure for CLP Pesticide Package

2.1 Standards Package

2.1.1 Forms

____ Form VIII - pages 1 & 2, and Form IX's for each run sequence are present. Form X present if compounds are reported.

____ % RSD < 10%. If over, proper corrective action was taken.

____ If % RSD > 10% for DDT only, DDE, DDD, DDT linearity series was run Graphs plotted if required.

____ % Breakdown for DDT and Endrin ≤ 20%.

____ Proper analytical sequence.

____ % Difference for Dibutyl Chlorendate ≤ 2%.

____ Any data not included in case has EPA SMO ID of ZZZZZ.

____ Documentation concerning the analytical sequence as required in the narrative.

____ Any corrections on forms are initialed and dated.

____ Make needed corrections on forms, initial and date.

____ Verify presence of Form IX for quantitation standards listed on Form VIII.

____ % Difference for calibration factors ≤ 15% for quantitation and ≤ 20% for confirmation.

____ DDT retention time window is reported for each 72-hour analytical sequence.

____ Verify data system calculation of response factors.

____ Check computer input for linearity and calibration factor % difference calculations.

2.1.2 Chromatograms

____ Chromatograms labeled with Case No., sample ID, instrument, date and time of injection, column, and volume injected.

____ Standard peaks labeled with peak ID and nanograms injected.

____ Copies of AR1221 and AR1232 chromatograms included with the first sequence's chromatograms. Verify that these chromatograms are labeled with the correct case number or project code and that their run dates are within thirty days prior to sample analysis.

____ AR1221 and AR1232 must be analyzed once each 30 days, and each primary run sequence for sample analysis must fall within the 30-day period of calibration for those aroclors.

____ Verify presence and chronological order of chromatograms for standards listed in the analytical sequence on each Form VIII. Verify that dates and times of injection on Form VIII, page 1 (EVALB injections), and page 2 agree with the date and time found on the chromatogram.

____ Analyst has initialed and dated chromatogram at beginning of chromatogram and beside the chromatogram report.

____ Initial and date any corrections or additions made by data reviewer.

QC Summary

____ Forms II, III, and IV are present.

____ No HSL compounds found ≥ CRDL in blanks.

____ Surrogate % recoveries within suggested values. Verify results for any sample outside the window. (Check all calculations.)

____ Spike recoveries and RPD inside windows. If not, verify results. (Check identifications and all calculations.)

____ Forms filled out completely.

____ Proper form used - water/soil.

____ Any corrections are initialed and dated.

2.3 Sample Data and Raw QC Data

Check at least 20% of sample packages. Check all blank packages. A package consists of Form I, chromatograms, raw data sheet, calculation sheets/computer calculation sheets.

____ Form I filled out completely and correctly. Check sample type, date received, date extracted, date analyzed, dilution, sample volume or weight used, extraction level, extraction type, GPC clean-up, pH, and SMO # or client ID.

____ Dates, times, and dilutions on chromatograms match those listed on Form I.

____ All raw data and calculation sheets are initialed and dated.

____ Chromatograms are labeled with Case No., sample ID, instrument ID, column ID, date and time of injection, volume injected, and analyst's initials and date.

____ Chromatogram peaks - positive identifications are labeled with name of compound, either above the peak or on the data system report.

____ Remove unnecessary chromatograms and make corrections to all forms involved.

____ Verify identification of all peaks.

____ Check for confirmation of positive ID's on primary column.

____ Verify results on raw data sheet.

____ Verify input into computer - computer calculations.

____ Verify manual calculations.

____ Initial and date any corrections made.

____ Initial and date each chromatogram checked.

____ Initial and date raw data and calculation sheets checked.

____ Verify that results on Form I match results on calculation forms.

____ Footnotes are present when needed.

____ Form X filled out correctly with sample information.

____ Only 2 dilutions submitted for each sample or QC sample.

2.4 Overall Package Check

____ All forms completely filled out.

____ Separate sections in order.

____ Address any analytical problems or noncompliance of data in the case narrative. Explain any problems and document corrective action taken.

Date _____

Project Code _____

QC Analyst _____

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: Analysis of Semivolatile Samples by GC/MS Under the CLP Contract	SOP NO: ME870212R0 DATE INITIATED: 02/12/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 12

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
W. T. Gibson	Alyce K. Moore	2-13-87	Janet M. Jones	2-13-87

1.0 Purpose

1.1 This SOP details procedures followed by ITAS-Knoxville for the analysis of CLP HSL semivolatiles. The CLP contract is the primary SOP for this analysis and is the ultimate source in matters of question.

1.2 Samples and standards are to be chromatographed, calculated, and reported according to CLP contract protocol. Changes to the contract protocol will be implemented as they are made by EPA. This SOP documents ITAS's specific procedures for the analysis of HSL semivolatiles. EPA's and ITAS's forms for calculation and reporting of data are included.

2.0 GC/MS Analysis

2.1 The code numbers of the samples to be analyzed, along with location and specific client requests, are found in the project work folder.

2.2 Samples and standards are injected onto a bonded phase GC capillary column by splitless technique using a grab injector. Data is acquired by consecutive mass spectra of peaks eluting from the column. Each acquisition has a standard or ITAS sample number name which is recorded automatically on a GC/MS run log sheet for the particular instrument. The run logs are initialed by the operator for each run with comments added if appropriate.

2.3 Finnigan 4000 and 4500 instruments are presently employed for semivolatiles analysis. Instructions for their operation, other than as given in this SOP, will be found in the operator's manual, the INCOS reference manual, or the schematics reference manual.

2.0 GC/MS Analysis (continued)

2.4 In the acquisition of any sample or standard, the following information must be included: SMO sample and case number (for samples), injection volume, instrument ID, column description, and program.

2.5 The master sequence for analysis of any samples or blanks must follow this pattern:

Tuning compound (DFTPP)
20 µg/ml standard
50 µg/ml standard
80 µg/ml standard
120 µg/ml standard
160 µg/ml standard

Daily 50 µg/ml standard (including DFTPP)
Method blank (same matrix as samples)
Sample 1
Sample 2
etc.
Matrix spike
Matrix spike duplicate

2.5.1 The 20-160 µg/ml standards comprise an initial five-point calibration that must meet certain criteria (see Form VI) before any further analysis may proceed. These standards may be run in any order as long as they are run within 12 hours of injection in a valid tuning compound run. The tuning compound may be included in the 50 µg/ml standard and the 50 µg/ml standard run first in the five-point.

2.5.2 The tuning compound (DFTPP-Difluorotriphenylphosphine) is directly injected either above or as part of the 50 µg/ml standard such that 50 µg is placed on column. The spectral data from its elution is assayed. The spectrum obtained must meet certain criteria (Form V) before any further analysis may proceed.

2.5.3 The daily 50 µg/ml standard must compare to the initial five-point calibration (Form VII) before any further analysis may proceed.

2.5.4 The method blank must be that blank prepared (extracted) with the set of samples under analysis, and must show no undesirably high levels of target compounds, specifically no greater than five times the contract required detection limits (CRDL) of common phthalates. Other target compounds should be less than CRDL limits (see Exhibit C for CRDL). If the blank fails these

2.0 GC/MS Analysis (continued)

criteria, the samples extracted with that blank are invalid and the samples must be reextracted. For this reason, the method blank should be run as early as possible after extraction.

2.5.5 The daily standard, blanks, and samples are to be run within a twelve hour period which begins at injection of a valid tuning compound run. Several days' runs may be based on one initial five-point calibration as long as each daily standard compares as in 2.5.3 above and the instrument has not been altered. Otherwise, a new calibration is required.

2.5.6 Matrix spike samples are samples into which some target compounds are spiked to check for recovery. The prep lab keeps a log of how many samples have been run and prepares a matrix spike and duplicate extract at least once per 20 samples or more often depending on the project, as outlined in the project work folder.

2.6 Preliminary evaluation of samples and blanks:

In addition to the criteria noted in Section 2.1.4, samples and blanks must be monitored for surrogate recoveries and internal standard area stability and for saturation.

2.6.1 Surrogate recoveries for blanks must meet the criteria for that matrix (see Form II). Otherwise, samples based on that blank must be reextracted. It is reasonable to reanalyze a blank if it is felt errant recoveries may be due to technique or instrumental fault before returning the samples for reextraction. If the recovery is greater than 10%, one surrogate from the acid and base/neutral fraction of a sample run may exceed limits. Beyond that, the sample may be reanalyzed. If it still fails specs, the sample must be reextracted. The reextracted analysis data above is submitted if the new extract passes criteria; otherwise, both analyses are submitted as evidence of matrix effect.

2.6.2 Internal standard areas should hold within 50-200% of the areas of the daily standard within a given twelve hour period. If any sample or blank exceeds this range, the instrumentation must be inspected for malfunctions. When any problems are found and corrected, the sample or blank must be rerun.

2.6.3 In the event any target compound exceeds the range established by the five-point calibration or a target compound peak is saturated, the sample must be rerun at a higher dilution. Surrogate recoveries criteria may not apply as they are diluted out.

2.0 GC/MS Analysis (continued)

2.7 Any new instrument being brought on line for semivolatiles analysis must be evaluated for precision by running three standards at three-five times the CRDL (Exhibit C) and calculating the instrumental detection limit as three times the standard deviation of the quantitative results. This detection limit must, in all cases, be less than CRDL. The data is kept on file in the document coordinator's office.

3.0 Preparation of Volatile Standards, Blanks, and Samples

Preparations of blanks and samples must be done with all precautions (repeated rinsing of syringes with methylene chloride) against any contamination from previous samples or standards. All samples, standards, and blanks will contain 40 µg/ml of internal standard.

3.1 Standards Preparation

The Hazardous Substance List (HSL) standards used in the CLP analyses are prepared from Supelco and other suppliers' catalog stock. In all cases, the standards must be traceable to EPA standards available in the Quality Assurance Materials Bank, EMSL, Las Vegas, and EPA mixes are to be routinely prepared for comparison with other standards. All primary and secondary standards preparation is to be performed in the semivolatiles lab hood.

3.1.1 Logbook

Standards are numbered by consecutive code according to their concentration levels (primary or secondary) and date of preparation. This information, along with source, lot number, aliquots size, final volume, solvent used, and initial and final concentration, are entered in the semivolatile standards preparation logbook.

3.1.2 Primary standards for HSL semivolatiles are supplied by Supelco. Primary standards for matrix spikes are from the EPA Quality Assurance Materials Bank. The specific matrix spike standards used are listed on Form III.

3.1.3 Secondary standard mixes of HSL or matrix spike compounds are prepared by dilution of the standards in Section 3.1.2 into methylene chloride, or methanol for matrix spikes. The HSL concentration will be 200 µg/ml; for matrix spikes, the base/neutral species will be 100 µg/ml and acids at 200 µg/ml.

3.0 Preparation of Volatile Standards, Blanks, and Samples (continued)

3.1.4 Surrogate and internal standard mixes are prepared from the pure compounds as primary standards in methylene chloride. The internal standard solution for spiking is ultimately diluted to 4000 µg/ml, while the surrogates are diluted in methanol to produce levels of 100 µg/ml (base-neutral) and 200 µg/ml (acid) aliquots for spiking samples prior to extraction. The standards are:

Surrogates:	2-Fluorophenol
	Phenol-D5
	Nitrobenzene-D5
	2-Fluorobiphenyl
	Terphenyl-D14
	Tribromophenol
Internal Standards:	1,4-Dichlorobenzene-D5
	Naphthalene-D8
	Acenaphthene-D10
	Phenanthrene-D10
	Chrysene-D12
	Perylene-D12

3.1.5 Injection standards are prepared from dilution of the 200 µg/ml HSL standard mix and adding surrogates (in the 50 µg/ml standard) at 50-100 µg/ml. The internal standard is added at 40 µg/ml (ratio of 10 µl per ml) prior to analysis. Diluting solvent is methylene chloride.

3.1.6 Calibration Standards

Calibration standards may be prepared at these levels:

Calibration Std.	Int. Std. (µg/ml)	Surr. (µg/ml)	HSL (µg/ml)
20 µg/ml	40	-	20
50 µg/ml	40	50-100	50
80 µg/ml	40	-	80
120 µg/ml	40	-	120
160 µg/ml	40	-	160

These standards comprise the necessary five-point concentrations. The 50 µg/ml standard is used also as the daily calibration check standard.

3.0 Preparation of Volatile Standards, Blanks, and Samples (continued)

3.2 Method Blank Preparation

The method blank is prepared, if the matrix is water, by mixing 300 µl each of the BN and acid extracts, and adding 6 µl of the 4000 µg/ml internal standard. For a soil sample in which only one BNA extract is produced, 6 µl of the internal standard is added to 600 µl of the sample.

3.3 Sample preparation is the same as for the method blank by matrix.

4.0 Specific Instrument Parameters for Semivolatiles

4.1 Proper, consistent, documented instrumental conditions are required for the sample analyses. Much of the documentation is maintained automatically by the software.

4.1.1 Maintenance

The operator is expected (along with the maintenance technician if necessary) to perform daily, monthly, and quarterly maintenance on the instrument according to SOP No. M841219RO and to so indicate by initialing the spaces in the preventive maintenance logbook located at the GC/MS lab entrance. In addition, any more extensive maintenance is to be detailed, dated, and signed into the individual instrument repair and maintenance logbooks.

4.1.2 Tuning

The 4000 and 4500 are tuned manually by adjustment of potentiometers on the electronics module. When any tuning is performed, the parameters are recorded and dated on the tuning log maintained on the instrument. The log parameters are:

Emission current
Electron multiplier voltage
Electron energy
Quad offset at 69 and 414 AMU
Lens 1-5 settings

Tuning is accomplished by altering these parameters (and possibly adjusting pots on the RF/CD control board) to achieve a properly resolved FC43 and ultimately obtaining a spectrum of DFTPP which meets all criteria (see Form V). Refer to the operator's manual for details. Once an instrument is in tune for DFTPP, analysis may begin. Under no circumstances may any tuning adjustment be made during a twelve hour period without reanalyzing for DFTPP.

4.0 Specific Instrument Parameters for Semivolatiles (continued)

4.1.3 Calibration of the instrument means creating a valid calibration table. Acquire (using the parameters listed in Section 4.1.4 below) an FC43 spectrum and create a calibration table using the program "Cali". The supervisor or an experienced operator must evaluate the fit of the table produced.

4.1.4 DFTPP (tuning compound) analysis is performed with the following acquisition parameters:

Baseline	= 0
Minimum area	approximately 20
Fragment width	approximately 70
Sampling interval	200 usec
Peak width	2

The instrument is scanned at 35-500 AMU with 0.95 seconds up and .05 seconds hold time at bottom (1 second/scan).

50 ng of DFTPP is injected alone, or more commonly, as part of the daily standard, using the column program outlined below. The column used is a J&W DB-5, 25 meters, .32 mm ID, with 1 µ loading. The DFTPP usually elutes at around 1100 scans. A straightforward spectrum of the eluting peak is taken and must conform to Form V.

4.1.5 Standards, blanks, and samples analysis is performed with the same acquisition and scan parameters as given above. The typical GC program is:

3 minutes hold at 45°
10°/min to 325°
Grob split valve opens at 1 minute after injection
Acquisition begins at 1.5 minutes after injection
(Filament, multiplier on at acquisition).

4.1.5.1 Injection technique is important to maintain precision of the analysis. 1 or 2 µl of sample is drawn into the needle with about 0.5 µl of methylene chloride flushing solvent. The needle is injected smoothly through the septum until the syringe level buts against the injection surface. At 6 seconds the sample is injected rapidly (~ 1 second) and the needle immediately withdrawn.

4.0 Specific Instrument Parameters for Semivolatiles (continued)

4.1.6 Sample data is acquired for about 1800 scans, long enough to allow full elution of benzo(g,h,i)perylene.

Other standard instrument settings are:

Separator oven	-	320°
Manifold	-	100°
Electron energy	-	70 EV
Split flow	-	40 ml/min
Sweep flow	-	10 ml/min
Column flow	-	2 ml/min

Column end should be positioned at approximately 1 inch from the source.

5.0 Data System Operation and Specific Calculations and Interpretations by the Operator

5.1 ITAS uses a modified version of the Finnigan TCA procedure to obtain qualitative and quantitative data for target compounds. In essence, a reverse search of the library is done in the predicted window for each compound, and hits are predicted based on library match and retention time closest to a least square projection of probable scan. The hits and projected scans are then integrated. The resulting forms obtained from the procedure are:

RIC
Quan Report
Search Diagnostics
Log File Printout
Triple Spectra and Interpretation Sheets
Library Diagnostics

5.1.1 A copy of the Quan Report (included) indicates the specific compounds sought and the characteristic ions, along with the internal standards and surrogates and the data format. Calculation of amounts is based on the response factor (RF) from the daily standard. RF is defined as:

$$\frac{(\text{Area cpd})}{(\text{Area int std})} \qquad \frac{(\text{Conc'n int std})}{(\text{Conc'n compound})}$$

The Quan Report for the sample shows quantitated results for target compounds by the following relation:

$$\text{Conc'n cpd} = \text{conc'n int std} \frac{(\text{area cpd})}{(\text{area int std})} \quad \frac{(1)}{(RF)}$$

5.0 Data System Operation and Specific Calculations and Interpretations by the Operator (continued)

The correct RF's, retention times, and relative retention times on which to base a twelve hour series of runs is set by typing R; T; S in the Quan Report program for the daily standard.

5.1.2 Search diagnostics is a labeled printout of the file related scan list. It is to be used to interpret the quality of the data program and to determine if manual rechecking is needed. For example, if > 1 peak is seen in the search column, the operator should manually recheck to determine if the wrong peak was assigned. Also, the saturation column must immediately be checked for compounds outside the instrument range.

5.1.3 The log file printout must confirm that instrumental parameters are the same as those used for DFTPP, aside from column program.

5.1.4 The triple spectra (raw and enhanced, versus standard spectrum) sheets must be evaluated to see if qualitative criteria are achieved for target compounds, i.e.:

All peaks > 10% in standard are in sample spectrum.

All peaks agree standard-sample within 20% of base peak.

All peaks > 10% in sample are in standard spectrum or are accountable as background or interference.

Molecular peak should also be present.

The operator must make careful evaluation of the spectra and consult the supervisor if necessary before accepting or rejecting a marginal match.

5.1.5 The library diagnostics are a simplified, reduced printout of the overall sample results, primarily containing forward search library information for further confirmation of data. It is not to be used for quantitation of data; the Quan Report is the source for that.

5.2 For tentatively identified compounds, a procedural file is available that allows the operator to integrate all uninterfered internal standard peaks, after which other peaks are integrated and then calculated based on the following relation:

$$\text{concentration cpd, extract} = \text{concentration int std} \times \frac{\text{(peak height)}}{\text{(peak height nearest internal std)}}$$

5.0 Data System Operation and Specific Calculations and Interpretations by the Operator (continued)

The program automatically prints library spectral matches for the tentatively identified species. Forms ITAS-K-ME104R0 and ME105R0 indicate how final sample concentration is to be calculated.

5.2.1 Qualitative identification of tentatively identified compounds is based on the same criteria given in Section 5.1.4.

5.3 Standards forms for DFTPP, initial calibration, and continuing calibration can be generated by the data system through LIST or from MSDS response lists for the latter two. The use of these forms and others is explained in the next section.

6.0 Analysis Forms to be Filled Out by Operator

6.1 There are several forms developed either by the EPA or by ITAS which are to be correctly filled out by the GC/MS operator. In addition, project specific forms may be required. In general, the two basic types in use are CLP and commercial.

6.2 CLP forms must be used for all analyses under the present EPA contract. The CLP contract is the primary source of information on these forms. Any questions concerning them should be referred back to that contract. The operator must properly fill out those that pertain to his analysis; the forms may be related to QC or to analytical results.

6.2.1 QC forms include:

6.2.1.1 Form II, Surrogate Percent Recovery Summary. No semivolatiles surrogates should exceed the limits given (except for samples, in which one surrogate from each fraction may be out if > 10%) without rerun and confirmation of matrix effect.

6.2.1.2 Form III, Matrix Spike/Matrix Spike Duplicate Recovery

% recovery is calculated as:

$$\frac{(\text{conc MS} - \text{sample result})}{(\text{conc'n spike added})} \times 100$$

RPD (relative percent difference) is calculated as

$$2\,\frac{(\text{conc'n MS} - \text{conc MSD})}{(\text{conc MS} + \text{conc MSD})} \times 100$$

6.0 Analysis Forms to be Filled Out by Operator (continued)

6.2.1.3 Form IV, Method Blank Summary. Results must be presented to two significant figures (1 if less than 10). No semivolatile method blank may have more than the CRDL of HSL species, except phthalates may be up to five times the CRDL.

6.2.1.4 Form V, GC/MS Tuning and Mass Calibration. The "sample ID" must be the Sample Management Office (SMO) identification; the "lab ID" is the ITAS identification. Time of analysis must be entered as military time. DFTPP not meeting all stated criteria is unacceptable and samples based on it are invalid.

6.2.1.5 Form VI, Initial Calibration Data. The mean and relative standard deviation of all HSL compounds from the five-point calibration is to be calculated and presented. Calibration check compounds (CCC) may not exceed 30% RSD. System performance check compounds (SPCC) must have RF's greater than 0.05 for semivolatiles. This form is generated by ITAS software using the program "QRF".

6.2.1.6 Form VII, Continuing Calibration Check. The daily standard is evaluated for CCC and SPCC compounds. CCC % D may not exceed 25%.

% D (percent difference) is calculated as:

$$\left(\frac{\text{RF (5-point mean)} - \text{RF (daily standard)}}{\text{RF (5-point mean)}}\right) \times 100$$

All data for HSL's must be included.

6.2.2 Analytical CLP forms are OADS forms, which include both HSL and tentative ID data.

All header information must be properly filled out, including matrix, date of sample receipt, date of extraction, and percent moisture if applicable.

All data is to be entered with two significant figures (or one if less than ten) with appropriate use of qualifiers. All undetected compounds are represented with a U and their contract required detection limit. Values less than CRDL, estimated (tentative ID's) receive a J qualifier. Any compound seen in the blank as well receives a B qualifier.

The sample number is the SMO sample number.

6.0 Analysis Forms to be Filled Out by Operator (continued)

6.3 Commercial forms have been developed at ITAS for priority pollutant and other analyses; some have been superceded by CLP forms. Instructions concerning these forms are to be found in the memos on:

QA/QC requirements for commercial (non-CLP) analysis, and
Project Data Reports.

Sample Number

Organics Analysis Data Sheet
(Page 1)

Laboratory Name: _____ Case No: _____

Lab Sample ID No: _____ QC Report No: _____

Sample Matrix: _____ Contract No: _____

Data Release Authorized By: _____ Date Sample Received: _____

Volatile Compounds

Concentration: Low Medium (Circle One)

Date Extracted/Prepared: _____

Date Analyzed: _____

Conc/Dil Factor: _____ pH _____

Percent Moisture: (Not Decanted) _____

CAS Number		ug/l or ug/Kg (Circle One)
74-87-3	Chloromethane	
74-83-9	Bromomethane	
75-01-4	Vinyl Chloride	
75-00-3	Chloroethane	
75-09-2	Methylene Chloride	
67-64-1	Acetone	
75-15-0	Carbon Disulfide	
75-35-4	1, 1-Dichloroethene	
75-34-3	1, 1-Dichloroethane	
156-60-5	Trans-1, 2-Dichloroethene	
67-66-3	Chloroform	
107-06-2	1, 2-Dichloroethane	
78-93-3	2-Butanone	
71-55-6	1, 1, 1-Trichloroethane	
56-23-5	Carbon Tetrachloride	
108-05-4	Vinyl Acetate	
75-27-4	Bromodichloromethane	

CAS Number		ug/l or ug/Kg (Circle One)
78-87-5	1, 2-Dichloropropane	
10061-02-6	Trans-1, 3-Dichloropropene	
79-01-6	Trichloroethene	
124-48-1	Dibromochloromethane	
79-00-5	1, 1, 2-Trichloroethane	
71-43-2	Benzene	
10061-01-5	cis-1, 3-Dichloropropene	
110-75-8	2-Chloroethylvinylether	
75-25-2	Bromoform	
108-10-1	4-Methyl-2-Pentanone	
591-78-6	2-Hexanone	
127-18-4	Tetrachloroethene	
79-34-5	1, 1, 2, 2-Tetrachloroethane	
108-88-3	Toluene	
108-90-7	Chlorobenzene	
100-41-4	Ethylbenzene	
100-42-5	Styrene	
	Total Xylenes	

Data Reporting Qualifiers

For reporting results to EPA, the following results qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

— Value	If the result is a value greater than or equal to the detection limit, report the value
U	Indicates compound was analyzed for but not detected. Report the minimum detection limit for the sample with the U (e.g., 10U) based on necessary concentration/dilution action. (This is not necessarily the instrument detection limit.) The footnote should read: U-Compound was analyzed for but not detected. The number is the minimum attainable detection limit for the sample
J	Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or when the mass spectral data indicated the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero (e.g., 10J). If limit of detection is 10 µg/l and a concentration of 3 µg/l is calculated, report as 3J.
C	This flag applies to pesticide parameters where the identification has been confirmed by GC-MS. Single component pesticides ≥10 ng/ul in the final extract should be confirmed by GC-MS.
B	This flag is used when the analyte is found in the blank as well as a sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action
Other	Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the data summary report

Laboratory Name _____

Case No: _____

Sample Number

Organics Analysis Data Sheet
(Page 2)

Semivolatile Compounds

Concentration: Low Medium (Circle One)

Date Extracted/Prepared: _____

Date Analyzed: _____

Conc/Dil Factor: _____

Percent Moisture (Decanted) _____

GPC Cleanup ☐Yes ☐No

Separatory Funnel Extraction ☐Yes

Continuous Liquid - Liquid Extraction ☐Yes

CAS Number		ug/l or ug/Kg (Circle One)
108-95-2	Phenol	
111-44-4	bis(-2-Chloroethyl)Ether	
95-57-8	2-Chlorophenol	
541-73-1	1, 3-Dichlorobenzene	
106-46-7	1, 4-Dichlorobenzene	
100-51-6	Benzyl Alcohol	
95-50-1	1, 2-Dichlorobenzene	
95-48-7	2-Methylphenol	
39638-32-9	bis(2-chloroisopropyl)Ether	
106-44-5	4-Methylphenol	
621-64-7	N-Nitroso-Di-n-Propylamine	
67-72-1	Hexachloroethane	
98-95-3	Nitrobenzene	
78-59-1	Isophorone	
88-75-5	2-Nitrophenol	
105-67-9	2, 4-Dimethylphenol	
65-85-0	Benzoic Acid	
111-91-1	bis(-2-Chloroethoxy)Methane	
120-83-2	2, 4-Dichlorophenol	
120-82-1	1, 2, 4-Trichlorobenzene	
91-20-3	Naphthalene	
106-47-8	4-Chloroaniline	
87-68-3	Hexachlorobutadiene	
59-50-7	4-Chloro-3-Methylphenol	
91-57-6	2-Methylnaphthalene	
77-47-4	Hexachlorocyclopentadiene	
88-06-2	2, 4, 6-Trichlorophenol	
95-95-4	2, 4, 5-Trichlorophenol	
91-58-7	2-Chloronaphthalene	
88-74-4	2-Nitroaniline	
131-11-3	Dimethyl Phthalate	
208-96-8	Acenaphthylene	
99-09-2	3-Nitroaniline	

CAS Number		ug/l or ug/Kg (Circle One)
83-32-9	Acenaphthene	
51-28-5	2, 4-Dinitrophenol	
100-02-7	4-Nitrophenol	
132-64-9	Dibenzofuran	
121-14-2	2, 4-Dinitrotoluene	
606-20-2	2, 6-Dinitrotoluene	
84-66-2	Diethylphthalate	
7005-72-3	4-Chlorophenyl-phenylether	
86-73-7	Fluorene	
100-01-6	4-Nitroaniline	
534-52-1	4, 6-Dinitro-2-Methylphenol	
86-30-6	N-Nitrosodiphenylamine (1)	
101-55-3	4-Bromophenyl-phenylether	
118-74-1	Hexachlorobenzene	
87-86-5	Pentachlorophenol	
85-01-8	Phenanthrene	
120-12-7	Anthracene	
84-74-2	Di-n-Butylphthalate	
206-44-0	Fluoranthene	
129-00-0	Pyrene	
85-68-7	Butylbenzylphthalate	
91-94-1	3, 3'-Dichlorobenzidine	
56-55-3	Benzo(a)Anthracene	
117-81-7	bis(2-Ethylhexyl)Phthalate	
218-01-9	Chrysene	
117-84-0	Di-n-Octyl Phthalate	
205-99-2	Benzo(b)Fluoranthene	
207-08-9	Benzo(k)Fluoranthene	
50-32-8	Benzo(a)Pyrene	
193-39-5	Indeno(1, 2, 3-cd)Pyrene	
53-70-3	Dibenz(a, h)Anthracene	
191-24-2	Benzo(g, h, i)Perylene	

(1)-Cannot be separated from diphenylamine

Laboratory Name: _____

Case No: _____

Sample Number

Organics Analysis Data Sheet
(Page 4)

Tentatively Identified Compounds

	CAS Number	Compound Name	Fraction	RT or Scan Number	Estimated Concentration (ug/l or ug/kg)
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					
17.					
18.					
19.					
20.					
21.					
22.					
23.					
24.					
25.					
26.					
27.					
28.					
29.					
30.					

SOIL SURROGATE PER[illegible]IT RECOVERY SUMMARY

Case No. _____ Contract Laboratory _____ Contract No. _____

Low _____ Medium _____

	VOLATILE				SEMI-VOLATILE							PESTICIDE
SMO TRAFFIC NO.	TOLUENE-D8 (81-117)	BFB (74-121)	1,2 DICHLOROETHANE-D4 (70-121)	NITROBENZENE-D5 (23-120)	2-FLUOROBIPHENYL (30-115)	TERPHENYL-D14 (18-137)			PHENOL-D5 (24-113)	2-FLUOROPHENOL (25-121)	2,4,6 TRIBROMOPHENOL (19-122)	DIBUTYLCHLORENDATE ** (20-150)

* VALUES ARE OUTSIDE OF CONTRACT REQUIRED QC LIMITS

** ADVISORY LIMITS ONLY

Volatiles: _____ out of _____ ; outside of QC limits

Semi-Volatiles: _____ out of _____ ; outside of QC limits

Pesticides: _____ out of _____ ; outside of QC limits

7/85

Comments: _____

FORM II

WATER SURROGATE PERCENT RECOVERY SUMMARY

Case No. _____ Contract Laboratory _____ Contract No. _____

	VOLATILE			SEMI-VOLATILE								PESTICIDE
EMO TRAFFIC NO.	TOLUENE-D8 (88-110)	BFB (86-115)	1,2 DICHLOROETHANE-D4 (76-114)	NITROBENZENE-D5 (35-114)	2-FLUOROBIPHENYL (43-116)	TERPHENYL-D14 (33-141)			PHENOL-D5 (10-94)	2-FLUOROPHENOL (21-100)	2,4,6 TRIBROMOPHENOL (10-123)	DIBUTYLCHLORENDATE ** (24-154)

* VALUES ARE OUTSIDE OF CONTRACT REQUIRED QC LIMITS

** ADVISORY LIMITS ONLY

Volatiles: _____ out of _____ ; outside of QC limits

Semi-Volatiles: _____ out of _____ ; outside of QC limits

Pesticides: _____ out of _____ ; outside of QC limits

Comments: _____

ATER MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Case No. _____ **Contractor** _____ **Contract No.** _____

FRACTION	COMPOUND	CONC. SPIKE ADDED (ug/L)	SAMPLE RESULT	CONC. MS	% REC	CONC. MSD	% REC	RPD	QC LIMITS * RPD	QC LIMITS * RECOVERY
VOA SMO SAMPLE NO. _____	1,1-Dichloroethene								14	61-145
	Trichloroethene								14	71-120
	Chlorobenzene								13	75-130
	Toluene								13	76-125
	Benzene								11	76-127
B/N SMO SAMPLE NO. _____	1,2,4-Trichlorobenzene								28	39-98
	Acenaphthene								31	46-118
	2,4 Dinitrotoluene								38	24-96
	Pyrene								31	26-127
	N-Nitroso-Di-n-Propylamine								38	41-116
	1,4-Dichlorobenzene								28	36-97
ACID SMO SAMPLE NO. _____	Pentachlorophenol								50	9-103
	Phenol								42	12-89
	2-Chlorophenol								40	27-123
	4-Chloro-3-Methylphenol								42	23-97
	4-Nitrophenol								50	10-80
PEST SMO SAMPLE NO. _____	Lindane								15	56-123
	Heptachlor								20	40-131
	Aldrin								22	40-120
	Dieldrin								18	52-126
	Endrin								21	56-121
	4,4'-DDT								27	38-127

* ASTERISKED VALUES ARE OUTSIDE QC LIMITS.

RPD:
VOAs ____ out of ____ : outside QC limits
B/N ____ out of ____ : outside QC limits
ACID ____ out of ____ : outside QC limits
PEST ____ out of ____ : outside QC limits

RECOVERY:
VOAs ____ out of ____ : outside QC limits
B/N ____ out of ____ : outside QC limits
ACID ____ out of ____ : outside QC limits
PEST ____ out of ____ : outside QC limits

Comments: _____

SOIL MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Case No. _____ Contractor _____ Contract No. _____

Low Level _____ Medium Level _____

FRACTION	COMPOUND	CONC. SPIKE ADDED (ug/Kg)	SAMPLE RESULT	CONC. MS	% REC	CONC. MSD	% REC	RPD	QC LIMITS * RPD	QC LIMITS * RECOVERY
VOA SMO SAMPLE NO. _____	1,1-Dichloroethene								22	59-172
	Trichloroethene								24	62-137
	Chlorobenzene								21	60-133
	Toluene								21	59-139
	Benzene								21	66-142
B/N SMO SAMPLE NO. _____	1,2,4-Trichlorobenzene								23	38-107
	Acenaphthene								19	31-137
	2,4 Dinitrotoluene								47	28-89
	Pyrene								36	35-142
	N-Nitrosodi-n-Propylamine								38	41-126
	1,4-Dichlorobenzene								27	28-104
ACID SMO SAMPLE NO. _____	Pentachlorophenol								47	17-109
	Phenol								35	26-90
	2-Chlorophenol								50	25-102
	4-Chloro-3-Methylphenol								33	26-103
	4-Nitrophenol								50	11-114
PEST SMO SAMPLE NO. _____	Lindane								50	46-127
	Heptachlor								31	35-130
	Aldrin								43	34-132
	Dieldrin								38	31-134
	Endrin								45	42-139
	4,4'-DDT								50	23-134

*ASTERISKED VALUES ARE OUTSIDE QC LIMITS.

RPD: VOAs ____ out of ____ ; outside QC limits
B/N ____ out of ____ ; outside QC limits
ACID ____ out of ____ ; outside QC limits
PEST ____ out of ____ ; outside QC limits

RECOVERY: VOAs ____ out of ____ ; outside QC limits
B/N ____ out of ____ ; outside QC limits
ACID ____ out of ____ ; outside QC limits
PEST ____ out of ____ ; outside QC limits

Comments: _____

METHOD _ ANK SUMMARY

Case No. _____ Region _____ Contractor _____ Contract No. _____

FILE ID	DATE OF ANALYSIS	FRACTION	MATRIX	CONC. LEVEL	INST. ID	CAS NUMBER	COMPOUND (HSL, TIC OR UNKNOWN)	CONC.	UNITS	CRQL

Comments: _____

FORM IV 7/85

GC/MS TUNING AND MASS CALIBRATION

Bromofluorobenzene (BFB)

Case No. _____ Contractor _____ Contract No. _____

Instrument ID _____ Date _____ Time _____

Lab ID _____ Data Release Authorized By: _____

m/e	ION ABUNDANCE CRITERIA	%RELATIVE ABUNDANCE
50	15.0 - 40.0% of the base peak	
75	30.0 - 60.0% of the base peak	
95	Base peak, 100% relative abundance	
96	5.0 - 9.0% of the base peak	
173	Less than 1.0% of the base peak	
174	Greater than 50.0% of the base peak	
175	5.0 - 9.0% of mass 174	( )[1]
176	Greater than 95.0%, but less than 101.0% of mass 174	( )[1]
177	5.0 - 9.0% of mass 176	( )[2]

THIS PERFORMANCE TUNE APPLIES TO THE FOLLOWING SAMPLES, BLANKS AND STANDARDS.

[1]Value in parenthesis is % mass 174.
[2]Value in parenthesis is % mass 176.

SAMPLE ID	LAB ID	DATE OF ANALYSIS	TIME OF ANALYSIS

Initial Calibration Data
Volatile HSL Compounds

Case No: _____ Region: _____ Instrument I D: _____

Contractor: _____ Calibration Date: _____

Contract No: _____

Minimum $\overline{RF}$ for SPCC is 0.300 (0.25 for Bromoform) **Maximum % RSD for CCC is 30%**

Laboratory ID								
Compound	RF_{20}	RF_{50}	RF_{100}	RF_{150}	RF_{200}	$\overline{RF}$	% RSD	CCC* SPCC**
Chloromethane								**
Bromomethane								
Vinyl Chloride								*
Chloroethane								
Methylene Chloride								
Acetone								
Carbon Disulfide								
1, 1-Dichloroethene								*
1, 1-Dichloroethane								**
Trans-1, 2-Dichloroethene								
Chloroform								*
1, 2-Dichloroethane								
2-Butanone								
1, 1, 1-Trichloroethane								
Carbon Tetrachloride								
Vinyl Acetate								
Bromodichloromethane								
1, 2-Dichloropropane								*
Trans-1, 3-Dichloropropene								
Trichloroethene								
Dibromochloromethane								
1, 1, 2-Trichloroethane								
Benzene								
cis-1, 3-Dichloropropene								
2-Chloroethylvinylether								
Bromoform								**
4-Methyl-2-Pentanone								
2-Hexanone								
Tetrachloroethene								
1, 1, 2, 2-Tetrachloroethane								**
Toluene								*
Chlorobenzene								**
Ethylbenzene								*
Styrene								
Total Xylenes								

RF -Response Factor (subscript is the amount of ug/L)
$\overline{RF}$ -Average Response Factor
%RSD -Percent Relative Standard Deviation

CCC -Calibration Check Compounds (*)
SPCC -System Performance Check Compounds (**)

Form VI

Continuing Calibration Check
Volatile HSL Compounds

Case No: _____ Region: _____ Calibration Date: _____

Contractor: _____ Time: _____

Contract No: _____ Laboratory ID: _____

Instrument ID: _____ Initial Calibration Date: _____

Minimum RF for SPCC is 0.300 (0.25 for Bromoform)

Maximum %D for CCC is 25%

Compound	RF	RF_{50}	% D	CCC	SPCC
Chloromethane					••
Bromomethane					
Vinyl Chloride				•	
Chloroethane					
Methylene Chloride					
Acetone					
Carbon Disulfide					
1, 1-Dichloroethene				•	
1, 1-Dichloroethane					••
Trans-1, 2-Dichloroethene					
Chloroform				•	
1, 2-Dichloroethane					
2-Butanone					
1, 1, 1-Trichloroethane					
Carbon Tetrachloride					
Vinyl Acetate					
Bromodichloromethane					
1, 2-Dichloropropane				•	
Trans-1, 3-Dichloropropene					
Trichloroethene					
Dibromochloromethane					
1, 1, 2-Trichloroethane					
Benzene					
cis-1, 3-Dichloropropene					
2-Chloroethylvinylether					
Bromoform					••
4-Methyl-2-Pentanone					
2-Hexanone					
Tetrachloroethene					
1, 1, 2, 2-Tetrachloroethane					••
Toluene				•	
Chlorobenzene					••
Ethylbenzene				•	
Styrene					
Total Xylenes					

RF_{50} -Response Factor from daily standard file at 50 ug/l
RF -Average Response Factor from initial calibration Form VI

%D -Percent Difference
CCC -Calibration Check Compounds (•)
SPCC -System Performance Check Compounds (••)

EXHIBIT C

Hazardous Substance List (HSL) and
Contract Required Detection Limits (CRDL)**

Volatiles	CAS Number	Detection Limits* Low Water[a] ug/L	Low Soil/Sediment[b] ug/Kg
1. Chloromethane	74-87-3	10	10
2. Bromomethane	74-83-9	10	10
3. Vinyl Chloride	75-01-4	10	10
4. Chloroethane	75-00-3	10	10
5. Methylene Chloride	75-09-2	5	5
6. Acetone	67-64-1	10	10
7. Carbon Disulfide	75-15-0	5	5
8. 1,1-Dichloroethene	75-35-4	5	5
9. 1,1-Dichloroethane	75-35-3	5	5
10. trans-1,2-Dichloroethene	156-60-5	5	5
11. Chloroform	67-66-3	5	5
12. 1,2-Dichloroethane	107-06-2	5	5
13. 2-Butanone	78-93-3	10	10
14. 1,1,1-Trichloroethane	71-55-6	5	5
15. Carbon Tetrachloride	56-23-5	5	5
16. Vinyl Acetate	108-05-4	10	10
17. Bromodichloromethane	75-27-4	5	5
18. 1,1,2,2-Tetrachloroethane	79-34-5	5	5
19. 1,2-Dichloropropane	78-87-5	5	5
20. trans-1,3-Dichloropropene	10061-02-6	5	5
21. Trichloroethene	79-01-6	5	5
22. Dibromochloromethane	124-48-1	5	5
23. 1,1,2-Trichloroethane	79-00-5	5	5
24. Benzene	71-43-2	5	5
25. cis-1,3-Dichloropropene	10061-01-5	5	5

(continued)

Volatiles	CAS Number	Detection Limits* Low Water[a] ug/L	Low Soil/Sediment[b] ug/Kg
26. 2-Chloroethyl Vinyl Ether	110-75-8	10	10
27. Bromoform	75-25-2	5	5
28. 2-Hexanone	591-78-6	10	10
29. 4-Methyl-2-pentanone	108-10-1	10	10
30. Tetrachloroethene	127-18-4	5	5
31. Toluene	108-88-3	5	5
32. Chlorobenzene	108-90-7	5	5
33. Ethyl Benzene	100-41-4	5	5
34. Styrene	100-42-5	5	5
35. Total Xylenes		5	5

[a]Medium Water Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Water CRDL.

[b]Medium Soil/Sediment Contract Required Detection Limits (CRDL) for Volatile HSL Compounds are 100 times the individual Low Soil/Sediment CRDL.

QUANTITATION REPORT FILE: VSTD01291

DATA: VSTD01291.TI
01/29/87 9:52:00
SAMPLE: 20PPB CLP CAL STANDARD (V0059)
UNDS.: *INST-OWA3* EPA CLP METHOD (PURGEABLES)
ORMULA: WATER INSTRUMENT: OWA3 WEIGHT: 0.000
SUBMITTED BY: ANALYST: TOWERY ACCT. NO.:
AMOUNT=AREA * REF. AMNT/(REF. AREA)* RESP. FACT); DET. LIM. = 0.00
RESP. FAC. FROM LIBRARY ENTRY

NO	NAME
1	(I.S. #1) BROMOCHLOROMETHANE
2	(I.S. #2) 1,4-DIFLUOROBENZENE
3	(I.S. #3) CHLOROBENZENE-D5
4	(S.S. #1) 1,2-DICHLOROETHANE-D4
5	(S.S. #2) TOLUENE-D8
6	(S.S. #3) 4-BROMOFLUOROBENZENE
7	CHLOROMETHANE **
8	BROMOMETHANE
9	VINYL CHLORIDE
10	CHLOROETHANE
11	METHYLENE CHLORIDE
12	ACETONE
13	CARBON DISULFIDE
14	1,1-DICHLOROETHENE *
15	1,1-DICHLOROETHANE **
16	TRANS-1,2-DICHLOROETHENE
17	CHLOROFORM *
18	1,2-DICHLOROETHANE
9	2-BUTANONE
20	1,1,1-TRICHLOROETHANE
21	CARBON TETRACHLORIDE
22	VINYL ACETATE
23	BROMODICHLOROMETHANE
24	1,2-DICHLOROPROPANE *
25	TRANS-1,3-DICHLOROPROPENE
26	TRICHLOROETHENE
27	DIBROMOCHLOROMETHANE
28	1,1,2-TRICHLOROETHANE
29	BENZENE
30	CIS-1,3-DICHLOROPROPENE
31	2-CHLOROETHYL VINYL ETHER
32	BROMOFORM **
33	2-HEXANONE
34	4-METHYL-2-PENTANONE
35	TETRACHLOROETHENE
36	1,1,2,2-TETRACHLOROETHANE **
37	TOLUENE
38	CHLOROBENZENE
39	ETHYL BENZENE *
40	STYRENE
41	TOTAL XYLENES

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA(HGHT)	AMOUNT	%TOT
1	128	327	10:54	1	1.000	A BB	30642.	50.000 UG/L	5.00
2	114	671	22:22	2	1.000	A BB	149162.	50.000 UG/L	5.00
3	117	824	27:28	3	1.000	A BB	127421.	50.000 UG/L	5.00

WGT
1-29-87

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA(HGHT)	AMOUNT	%TOT
4	65	420	14:00	1	1.284	A BB	55509.	50.000 UG/L	5.00
5	98	789	26:18	3	0.958	A BB	128681.	50.000 UG/L	5.00
6	95	975	32:30	3	1.183	A BB	99799.	50.000 UG/L	5.00
7	50	54	1:48	1	0.165	A BB	15462.	20.000 UG/L	2.00
8	94	86	2:52	1	0.263	A BB	16085.	20.000 UG/L	2.00
9	62	114	3:48	1	0.349	A BB	12406.	20.000 UG/L	2.00
10	64	148	4:56	1	0.453	A BB	7268.	20.000 UG/L	2.00
11	84	219	7:18	1	0.670	A BB	12537.	20.000 UG/L	2.00
12	58	256	8:32	1	0.783	A BB	630.	20.000 UG/L	2.00
13	76	284	9:28	1	0.869	A BB	21806.	20.000 UG/L	2.00
14	96	322	10:44	1	0.985	A BB	11989.	20.000 UG/L	2.00
15	63	359	11:58	1	1.098	A BB	20968.	20.000 UG/L	2.00
16	96	388	12:56	1	1.187	A BV	12297.	20.000 UG/L	2.00
17	83	394	13:08	1	1.205	A BB	27991.	20.000 UG/L	2.00
18	62	424	14:08	1	1.297	A BB	24535.	20.000 UG/L	2.00
19	72	430	14:20	2	0.641	A BB	1271.	20.000 UG/L	2.00
20	97	462	15:24	2	0.689	A BB	21049.	20.000 UG/L	2.00
21	117	475	15:50	2	0.708	A VB	23217.	20.000 UG/L	2.00
22	86	490	16:20	2	0.730	A BB	1468.	20.000 UG/L	2.00
23	83	488	16:16	2	0.727	A BB	23794.	20.000 UG/L	2.00
24	63	539	17:58	2	0.803	A BB	13231.	20.000 UG/L	2.00
25	75	547	18:14	2	0.815	A VB	17910.	16.000 UG/L	1.60
26	130	565	18:50	2	0.842	A BB	26982.	20.000 UG/L	2.00
27	129	575	19:10	2	0.857	A BB	23167.	20.000 UG/L	2.00
28	97	583	19:26	2	0.869	A BB	17159.	20.000 UG/L	2.00
29	78	591	19:42	2	0.881	A BB	36935.	20.000 UG/L	2.00
30	75	588	19:36	2	0.876	A BB	9995.	24.000 UG/L	2.40
31	63	625	20:50	2	0.931	A BB	7210.	20.000 UG/L	2.00
32	173	659	21:58	2	0.982	A BB	25052.	20.000 UG/L	2.00
33	100	741	24:42	3	0.899	A BB	1555.	20.000 UG/L	2.00
34	100	690	23:00	3	0.837	A BB	2684.	20.000 UG/L	2.00
35	164	744	24:48	3	0.903	A BB	34474.	20.000 UG/L	2.00
36	85	734	24:28	3	0.891	A BB	20976.	20.000 UG/L	2.00
37	92	795	26:30	3	0.965	A BB	30761.	20.000 UG/L	2.00
38	112	828	27:36	3	1.005	A BB	49238.	20.000 UG/L	2.00
39	106	896	29:52	3	1.087	A BB	22454.	20.000 UG/L	2.00
40	104	1036	34:32	3	1.257	A BB	41291.	20.000 UG/L	2.00
41	106	1078	35:56	3	1.308	A BB	26425.	20.000 UG/L	2.00

NO	RET(L)	RATIO	RRT(L)	RATIO	AMNT	AMNT(L)	R.FAC	R.FAC(L)	RATIO
1	10:54	1.00	1.000	1.00	50.00	50.00	1.000	1.000	1.00
2	22:22	1.00	1.000	1.00	50.00	50.00	1.000	1.000	1.00
3	27:28	1.00	1.000	1.00	50.00	50.00	1.000	1.000	1.00
4	14:00	1.00	1.284	1.00	50.00	50.00	1.812	1.812	1.00
5	26:18	1.00	0.958	1.00	50.00	50.00	1.010	1.010	1.00
6	32:30	1.00	1.183	1.00	50.00	50.00	0.783	0.783	1.00
7	1:48	1.00	0.165	1.00	20.00	20.00	1.262	1.262	1.00
8	2:52	1.00	0.263	1.00	20.00	20.00	1.312	1.312	1.00
9	3:48	1.00	0.349	1.00	20.00	20.00	1.012	1.012	1.00
10	4:56	1.00	0.453	1.00	20.00	20.00	0.593	0.593	1.00
11	7:18	1.00	0.670	1.00	20.00	20.00	1.023	1.023	1.00
12	8:32	1.00	0.783	1.00	20.00	20.00	0.051	0.051	1.00
13	9:28	1.00	0.869	1.00	20.00	20.00	1.779	1.779	1.00
14	10:44	1.00	0.985	1.00	20.00	20.00	0.946	0.946	1.00
15	11:58	1.00	1.098	1.00	20.00	20.00	1.678	1.678	1.00
16	12:56	1.00	1.187	1.00	20.00	20.00	1.003	1.003	1.00

WET
1-29-87

17	13:08	1.00	1.205	1.00	20.00	20.00	2.251	2.251	1.00
18	14:08	1.00	1.297	1.00	20.00	20.00	2.002	2.002	1.00
19	14:20	1.00	0.641	1.00	20.00	20.00	0.021	0.021	1.00
20	15:24	1.00	0.689	1.00	20.00	20.00	0.353	0.353	1.00
21	15:50	1.00	0.708	1.00	20.00	20.00	0.389	0.389	1.00
22	16:20	1.00	0.730	1.00	20.00	20.00	0.025	0.025	1.00
23	16:16	1.00	0.727	1.00	20.00	20.00	0.399	0.399	1.00
24	17:58	1.00	0.803	1.00	20.00	20.00	0.222	0.222	1.00
25	18:14	1.00	0.815	1.00	16.00	16.00	0.367	0.367	1.00
26	19:50	1.00	0.842	1.00	20.00	20.00	0.452	0.452	1.00
27	19:10	1.00	0.857	1.00	20.00	20.00	0.388	0.388	1.00
28	19:26	1.00	0.869	1.00	20.00	20.00	0.288	0.288	1.00
29	19:42	1.00	0.881	1.00	20.00	20.00	0.619	0.619	1.00
30	19:36	1.00	0.876	1.00	24.00	24.00	0.140	0.140	1.00
31	20:50	1.00	0.931	1.00	20.00	20.00	0.121	0.121	1.00
32	21:58	1.00	0.982	1.00	20.00	20.00	0.420	0.420	1.00
33	24:42	1.00	0.899	1.00	20.00	20.00	0.031	0.031	1.00
34	23:00	1.00	0.837	1.00	20.00	20.00	0.053	0.053	1.00
35	24:48	1.00	0.903	1.00	20.00	20.00	0.676	0.676	1.00
36	24:28	1.00	0.891	1.00	20.00	20.00	0.412	0.412	1.00
37	26:30	1.00	0.965	1.00	20.00	20.00	0.604	0.604	1.00
38	27:36	1.00	1.005	1.00	20.00	20.00	0.966	0.966	1.00
39	29:52	1.00	1.087	1.00	20.00	20.00	0.441	0.441	1.00
40	34:32	1.00	1.257	1.00	20.00	20.00	0.810	0.810	1.00
41	35:56	1.00	1.308	1.00	20.00	20.00	0.518	0.518	1.00

WET
1-29-87

EQUATION FOR QUANTIFYING TENTATIVELY IDENTIFIED COMPOUNDS

$$X = (C_{IS})\left(\frac{H_x}{H_{IS}}\right)\left(\frac{V_t}{V_o}\right) \quad (D)$$

$X\left(^{ug}/_{l}\right)$ = estimated concentration of tentatively identified compound

H_x = total ion current peak height of tentatively identified compound

H_{IS} = total ion current peak height of uninterfered internal standard nearest to tentatively identified compound

40. $= C_{IS}\left(^{ug}/_{ml}\right)$ = concentration of internal standard in sample extract

$= V_t\ (ml)$ = volume of total extract

$= V_o\ (l)$ = volume of water extracted

$= D$ = dilution factor

Laboratory Name: I. T. A. S. - Knoxville

Project Number : _____

Sample Number : _____

EQUATION FOR QUANTIFYING TENTATIVELY IDENTIFIED COMPOUNDS

$$X = (C_{IS})\left(\frac{H_x}{H_{IS}}\right)\left(\frac{V_t}{W_t \; D_R}\right) \quad (D)$$

$X\left(^{ug}/_{kg}\right)$ = estimated concentration of tentatively identified compound

$40. = C_{IS}\left(^{ug}/_{ml}\right)$ = concentration of internal standard in sample extract

H_x = total ion current peak height of tentatively identified compound

H_{IS} = total ion current peak height of uninterfered internal standard nearest to tentatively identified compound

$= V_t\,(ml)$ = volume of total extract

$= W_t(kg)$ = wet weight of soil extracted

$= D_R$ = dryness factor = $\frac{\text{sample dry weight}}{\text{sample wet weight}}$

$= D$ = dilution factor

MEDIUM PREP LOW PREP

Laboratory Name: I. T. A. S. - Knoxville

Project Number : _____

Sample Number : _____

Memo

To: All GC/MS personnel

From: Snell Mills

Subject: QA/QC requirements for commercial(non-CLP) analysis

In order to comply with QA/QC requirements as documented in the ITAS Quality Assurance Manual, I am implementing the following standard operating procedur Effective immediately all analysis will be performed according to the current EPA CLP statement of work with the following exceptions:

a) Only Priority Pollutant compounds will be reported (using current report forms) unless otherwise requested by client.

b) Tentatively identified compounds will not be required unless requested by client.

c) Matrix Spike / Matrix Spike Duplicate analysis will be done at a frequency five percent (5%) of all samples analyzed instead of a project(case) basis.

d) Initial Calibration Data is not required to be submitted with every report; however it must be retained on file by instrument/date analyzed. Continuing Calibration Data and Method Blank Data must be submitted with every project report. If the calibration and blank data pertains to more than one project a copy must be included with each project report.

e) Method Blank Summary Forms will not be required.

The following forms have been developed to be used for commercial reporting those items required under the CLP protocols:

1) Surrogate Recovery - Acid,B/N, & VOA fractions, both Soil & Water matrices

2) MS/MSD Recovery - Acid,B/N, & VOA fractions, both Soil & Water matrices

3) MS/MSD Sample prep

I realize that there will be certain situations and problems that cannot be solved within the CLP guidelines. These will have to be handled on a case by case basis. Any deviations from the CLP QA/QC criteria must be approved by the Group Leader and the QA/QC coordinator and be fully documented.

If you have any questions regarding this please feel free to ask for clarification..

MEMO

To: All GC/MS Personnel

From: Steve Lowry

Subject: Project Data Reports

Attached you will find guidelines by which all GC/MS raw data packages should be reported. We now have five people submitting raw data for review and we have two people reviewing data for release to the clients. Each person has, up to now, had their own way in which to sort the various pieces of raw data for submission and it has been very tiring to review projects for which several people have submitted data. Generally, everyone has had all the pieces present, but it is difficult to review data when all those pieces are in different places. The attached method of sorting the data follows CLP protocol except for the frount two pages. This allows the data rewiewers to quickly and efficiently check the pertainant pieces of data.

Also attached, you will find a copy of an earlier memo from Snell concerning the QA/QC requirements for commercial (non-CLP) analyses. This is included to give everyone a second look in hopes of tying all the data for a project together.

Everyone has been doing an outstanding job in keeping up with the tremendous work load under which we have been operating and getting good data into the review process.

If you have any questions or suggestions please let me know.

1 Summary

This document describes the procedure by which all GC/MS raw data should be orginized for submission for review. Raw data packages submitted by various GC/MS personnel should be in the same order so as to allow the more efficient data review.

2 Organization of Raw Data

2.1 I.T.A.S. Library Search Report

The I.T. Analytical Services Library Search Report must begin each raw data package. Any calculation involving dryness factors, dilution factors, densities, etc. must appear on this page in a general formula. Any references to other samples or dilutions or other comments must be noted. This page is required for all samples, blanks, duplicates, matrix spikes, and dilutions.

2.2 I.T.A.S. QA/QC Report

The I.T. Analytical Services QA/QC Report must be the second page. Any internal standards that are out of specifications must be commented as to corrective action taken or rationale for the problems encountered. Any Surrogate standard that is out of specifications must be noted.

2.3 Chromatogram

The chromatogram must be the third page of the raw data package. Any labling of peaks that is required (i.e. CLP data or client request for data, etc.) must be present.

2.4 Quantitation Report

The Quantitation Report must be present and reported with respect to the proper daily standard. The table of response factors must be present. All entries in the quantitation list must be listed with no detection limit set. Any deletion of entries must be by marking through unacceptable entries and initialing and dating the result sheet.

2.5 Instrument Parameters

The instrument parameters must be taken from the acquisition log file when it is present.

2.6 I.T. Analytical Services Diagnostic Report

The I.T.A.S. Diagnostic Report must be included immediately preceding the spectra. If it is not available, a page with comments as to why it is not must be included.

2.7 HSL/PP Spectra

All spectra for each Hazardous Substance List or Priority Pollutant List compound listed in the quantitation list must be included, immediately preceeded by a tabular representation of the library comparison. If the raw data is that of a dilution of a sample that is also reported as a more concentrated analysis then it is allowable to submit only the spectra of the components for which the raw data is valid (i.e. components which are within the calibration range). The spectra and listings are not required for matrix spike or matrix spike duplicate analyses.

3 Conclusion

All raw data submitted for review must meet these organizational criteria. Sample raw data submitted as part of a CLP data package must place in the order specified by the CLP contract. Commercial sample raw data must be stapled and submitted as part of a commercial project data package. Any deviations from this form must be either requested by the group supervisor as a special project or be cleared, signed, and dated by a group supervisor.

MEMO

To: All GC/MS Personnel

From: Steve Lowry

Subject: Sample Data Reports

Several items concerning sample data reports need to be addressed:

1. In the space provided for Lab Sample Number give only the sample number, not the data file name, unless the sample is a spiked dupolicate (i.e. X1111, X1111MS,& X1111MSD), an unnumbered duplicate (X1111 & X1111D), or is in some other way associated with another reported sample of the same lab sample number. All concentrations of a single sample should be collected on a single report form and a single lab sample number used.

2. In the space provided for the Sample I.D. give the full identification for the sample as given in the project data attached to each project worksheet. Give the I.D. "No ID" only if that is the I.D. given in the project data: If a sample has been given RUSH status and the numbers reported before a sample ID is determined leave the space blank or use a pencil to write in preliminary information.

3. All values greater than 10 ppb should be given to two (2) significant figures and all values less than 10 ppb should be given to one (1) significant figure, unless otherwise requested (i.e. 25ppb, 310ppm, .022ppm, .007ppm, 4.ppb, etc.).

4. All values less than the detection limit should be put in parentheses after the < DL (i.e. <10 (2), <1.0 (.050)).

5. Only one name should appear in the space provided for Reported by unless two people actually filled in reported values, in which case each person should sign their own

Please [redacted] any questions or provide any suggestions that you might [redacted]

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE:	SOP NO: MV870212R0 DATE INITIATED: 02/12/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 11
Analysis of Volatile Compounds by GC/MS Under the CLP Contract	

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
W. T. Wilson	Alyce L. Morse	2-13-87	Janet M. Jones	2-13-87

1.0 Purpose

1.1 This SOP details procedures followed by ITAS-Knoxville for the analysis of CLP HSL volatiles. The CLP contract is the primary SOP for this analysis and is the ultimate source in matters of question.

1.2 Samples and standards are to be chromatographed, calculated, and reported according to CLP contract protocol. Changes to the contract protocol will be implemented as they are made by EPA. This SOP documents ITAS's specific procedures for the analysis of HSL volatiles. EPA's and ITAS's forms for calculation and reporting of data are included.

2.0 GC/MS Analysis

2.1 The code numbers of the samples to be analyzed, along with location and specific client requests, are found in the project work folder.

2.2 Samples and standards are purged and trapped using a TEKMAR Liquid Sample Concentrator, then desorbed onto the GC column of the GC/MS instrument. Data is acquired by consecutive mass spectra of peaks eluting from the column. Each acquisition has a standard or ITAS sample number name which is recorded either manually or automatically on a GC/MS run log sheet for the particular instrument. The run logs are initialed by the operator for each run with comments added if appropriate.

2.3 Finnigan OWAs are presently employed for volatile analysis. Instructions for their operation, other than as given in this SOP, will be found in the operator's manual, the INCOS reference manual, or the schematics reference manual.

2.0 GC/MS Analysis (continued)

2.4 In the acquisition of any sample or standard, the following information must be included: SMO sample and case number (for samples), purge volume, instrument ID, column description, and program.

2.5 The master sequence for analysis of any samples or blanks must follow this pattern:

Tuning compound (BFB)
20 ppb standard
50 ppb standard
100 ppb standard
150 ppb standard
200 ppb standard

Tuning compound (BFB)
Daily 50 ppb standard
Method blank (same matrix as samples)
Sample 1
Sample 2
Matrix spike
Matrix spike duplicate

2.5.1 The 20-200 ppb standards comprise an initial five-point calibration that must meet certain criteria (see Form VI) before any further analysis may proceed. These standards may be run in any order as long as they are run within 12 hours of injection in a valid tuning compound run.

2.5.2 The tuning compound (BFB-P-Bromofluorobenzene) is directly injected at 50 ng on column and the spectral data from its elution is assayed. The spectrum obtained must meet certain criteria (see Form V) before any further analysis may proceed.

2.5.3 The daily 50 ppb standard must meet the QC criteria specified in Section E (see Form VII) before any further analysis may proceed.

2.5.4 The method blank must be representative of the matrix of the sample runs and must show no undesirably high levels of target compounds, specifically no greater than five times the contract required detection limits (CRDL) of common lab solvents such as methylene chloride, acetone, and toluene. Other target compounds must be less than CRDL limits (see Exhibit C for CRDL). If the blank fails these criteria, no further analyses may proceed. The daily blank is to be run prior to sample analyses.

2.0 GC/MS Analysis (continued)

2.5.5 The daily standard, blanks, and samples are to be run within a twelve hour period which begins at injection of a valid tuning compound run. Several days' runs may be based on one initial five-point calibration as long as each daily standard compares as in 2.5.3 above and the instrument has not been altered. Otherwise, a new calibration is required.

2.5.6 Matrix spike samples are samples into which some target compounds are spiked to check for recovery. The volatiles lab keeps a log of how many samples have been run and analyzes a matrix spike and duplicate at least once per 20 samples or more often depending on the project, as outlined in the project work folder.

2.6 Preliminary evaluation of samples and blanks:

In addition to the criteria noted in Section 2.1.4, samples and blanks must be monitored for surrogate recoveries and internal standard area stability and for saturation.

2.6.1 Surrogate recoveries for blanks must meet the criteria for that matrix (see Form II). Recoveries for samples may be due to matrix effects: if a sample run fails the criteria, a rerun is required; if the rerun meets the criteria, the first run data is rejected; if it fails, both runs' data is submitted as evidence of matrix effect.

2.6.2 Internal standard areas should hold within 50-200% of the areas of the daily standard within a given twelve hour period. If any sample or blank exceeds this range, the instrumentation must be inspected for malfunctions. When any problems are found and corrected, the sample or blank must be rerun.

2.6.3 In the event any target compound exceeds the range established by the five-point calibration or a target compound peak is saturated, the sample must be rerun at a higher dilution.

2.7 Any new instrument being brought on line for volatiles analysis must be evaluated for precision by running three standards at three-five times the CRDL (Exhibit C) and calculating the instrumental detection limit as three times the standard deviation of the quantitative results. This detection limit must, in all cases, be less than the CRDL. The data is kept on file in the document coordinator's office.

3.0 Preparation of Volatile Standards, Blanks, and Samples

All preparations are to be done in the volatiles laboratory to guard against contamination. Preparations of blanks and samples must be done with all precautions (repeated rinsing of microliter syringes with fresh methanol and ml syringes with reagent water) against any contamination from previous samples or standards. All samples, standards, and blanks contain 50 ppb of internal standard.

3.1 Standards Preparation

The Hazardous Substance List (HSL) standards used in the CLP analyses are prepared from Supelco and other suppliers' catalog stock. In all cases, the standards must be traceable to EPA standards available in the Quality Assurance Materials Bank, EMSL, Las Vegas, and EPA mixes are to be routinely prepared for comparison with other standards. All primary and secondary standards preparation is to be performed in the volatiles lab hood.

3.1.1 Logbook

Standards are numbered by consecutive code according to their concentration levels (primary or secondary) and date of preparation. This information, along with source, log number, aliquots size, final volume, solvent used, and initial and final concentration, are entered in the volatile standards preparation logbook.

3.1.2 Primary standards are prepared from pure stock of various suppliers for those compounds not supplied in Supelco purgeables A, B, and C. This includes matrix spike standards. The specific matrix spike standards used are listed on Form III. The solvent is purge and trap grade methanol.

3.1.3 Secondary HSL or matrix spike standard mixes are prepared by direct dilution of Supelco purgeables A, B, and C into purge and trap grade methanol, and by dilution of the primary standards from 3.1.2 above. The level should be at least 50 µg/ml. Specific HSL compounds for VOA's are listed in Exhibit C and example Quantitation Report.

3.1.4 Surrogate and internal standard mixes are prepared from Supelco stock as secondary standards. The standards are:

Surrogates:	1,2-Dichloroethane-D4
	Toluene-D8
	P-bromofluorobenzene
Internal Standards:	Bromochloromethane
	1,4-Difluorobenzene
	Chlorobenzene-D5

3.0 Preparation of Volatile Standards, Blanks, and Samples (continued)

3.1.5 Purge standards are the final form in which the standards are prepared and are made up just prior to analysis. Specifically, for the daily standard, the purge standard is prepared by injecting secondary HSL standards, along with surrogate and internal standards into 5.0 ml of reagent water (prepared by carbon filtration of deionized distilled water) in a gastight 5.0 ml luerlock syringe to produce a level of 50 ppb in the water. This standard is then ready to be introduced to the TEKMAR sample and purged.

3.1.6 Calibration Standards

In the same manner as the daily purge standard is prepared, calibration purge standards may be prepared at these levels:

Calibration Std.	Int. Std. (ppb)	Surr. (ppb)	HSL (ppb)
20 ppb	50	50	20
50 ppb	50	50	50
100 ppb	50	50	100
150 ppb	50	50	150
200 ppb	50	50	200

These standards, each one prepared just prior to analysis, comprise the necessary five-point calibrations.

3.2 Method Blank Preparation

Refer to Section 3.1.5. The method blank is prepared just as the daily standard except that no HSL standards are added. Additional "clean" purge and trap methanol may be added to the method blank to bring the methanol level in line with that in the sample.

3.3 Sample preparation depends on the matrix involved. Three matrices are defined: low water, low soil, and medium soil.

3.3.1 Low water samples are prepared by withdrawing 5.0 ml of sample into a 5 ml gastight luerlock syringe and injecting internal standard and surrogate solutions to 50 ppb in the aliquot. The sample is ready for purging. Should the sample require dilution, another 5 ml aliquot is taken at the same time and sealed in another gastight syringe from which dilutions may be made.

3.0 Preparation of Volatile Standards, Blanks, and Samples (continued)

3.3.2 Soil samples are prepared by weighing 5.0 grams of sample into the sampler vial (removed from the TEKMAR), and adding 5.0 ml of the method blank preparation. Up to 1/5 dilution may be achieved by using as little as 1.0 grams of soil.

3.3.3 Medium soil samples are prepared in the event that the low soil sample exceeds the instrument calibration range or saturates for some target compound. 4.0 grams of the sample is spiked with 1 ml of 25 µg/ml of surrogate solution, 9.0 ml of purge and trap methanol are added and mixed, and 100 µl of this extract is injected into 5.0 ml of reagent water in a gastight syringe prepared as for a method blank. This results in a 1/125 dilution sample ready for purging. Further dilutions can be achieved by injecting less than 100 ml into the reagent water.

4.0 Specific Instrument Parameters for Volatiles

4.1 Proper, consistent, documented instrumental conditions are required for the sample analyses. Much of the documentation is maintained automatically by the software.

4.1.1 Maintenance

The operator is expected (along with the maintenance technician if necessary) to perform daily, monthly, and quarterly maintenance on the instrument according to SOP No. M841219R0 and to so indicate by initialing the spaces in the preventive maintenance logbook located at the GC/MS lab entrance. In addition, any more extensive maintenance is to be detailed, dated, and signed into the individual instrument repair and maintenance logbooks.

4.1.2 Tuning

The OWA maintains its tuning parameters on disc to be accessed by the data system. Adjustment of lens, extractor, electron multiplier voltage, resolution, electrometer zero, and ion volume is achieved through the manual tune program and manual manipulation of the ion source magnet may also be required, with the goal of achieving a well resolved spectrum of FC43 and ultimately a spectrum of BFB which meets all criteria (see Form V). Refer to the operator's manual for details. Once an instrument is in tune for BFB, analysis may begin. Under no circumstance may any tuning adjustment be made during a twelve hour period without reanalyzing for BFB.

4.0 Specific Instrument Parameters for Volatiles (continued)

4.1.3 Calibration of the instrument means creating a valid calibration table. Acquire (using the parameters listed in Section 4.1.4 below) a FC43 spectrum and create a calibration table using the program "Cali". The supervisor or an experienced operator must evaluate the fit of the table produced.

4.1.4 BFB (tuning compound) analysis is performed with the following acquisition parameters:

Baseline	= 0
Minimum area	approximately 40
Fragment width	approximately 60
Sampling interval	200 µsec
Peak width	1-2

The instrument is scanned at 35-300 AMU with 1.95 seconds up and .05 seconds hold time at bottom (2 seconds/scan).

50 ng of BFB is injected at 230° isothermal. The column used is Supelco SP-1000. The acquisition is begun at injection; BFB usually elutes at around 400 scans. A straightforward spectrum of the eluting peak is taken and must conform to Form V.

4.1.5 Standards, blanks, and samples analysis is performed with the same acquisition and scan parameters as for BFB; however, the GC setting is changed to a hold time of 4 minutes at 50°, followed by a 10°/min program to 230°. The GC and acquisition programs begin in unison upon signal from the TEKMAR LSC-2 that desorption has begun. The TEKMAR sequence is as follows:

t = 0	purge begins at Purge Ready
t = 10 minutes	purge stops, trap heats to 180°
	Valve switches to Desorb
(at this stage GC program and acquisition begin)	
The TEKMAR sampler is rinsed at this time	
t = 14 minutes	Valve switches to bakeout position
t = 32 minutes	Bakeout at 220° complete, trap begins to cool to Purge Ready

Before purge begins, the purge standard, blank, or sample is introduced to the TEKMAR sampler through the luerlock fitting. The purged species are adsorbed on the tenax trap from which they are thermally desorbed at t = 10-14 minutes (4 minute desorb). During purge, the TEKMAR sampler (and sample) is heated to 40°C in a water bath.

4.0 Specific Instrument Parameters for Volatiles (continued)

Sample data is acquired for about 1200 scans, long enough to allow full elution of o-xylene.

Other standard instrument settings are:

Separator oven	-	250°
Manifold	-	90°
Electron energy	-	70 EV
Purge and column flow	-	40 ml/min

5.0 Data System Operation and Specific Calculations and Interpretations by the Operator

5.1 ITAS uses a modified version of the Finnigan TCA procedure to obtain qualitative and quantitative data for target compounds. In essence, a reverse search of the library is done in the predicted window for each compound, and hits are predicted based on library match and retention time closest to a least square projection of probable scan. The hits and projected scans are then integrated. The resulting forms obtained from the procedure are:

RIC
Quan Report
Search Diagnostics
Log File Printout
Triple Spectra and Interpretation Sheets
Library Diagnostics

5.1.1 A copy of the Quan Report (included) indicates the specific compounds sought and the characteristic ions, along with the internal standards and surrogates and the data format. Calculation of amounts is based on the response factor (RF) from the daily standard. RF is defined as:

$$\frac{(\text{Area cpd})}{(\text{Area int std})} \quad \frac{(\text{Conc'n int std})}{(\text{Conc'n compound})}$$

The Quan Report for the sample shows quantitated results for target compounds by the following relation:

$$\text{Conc'n cpd} = \text{conc'n int std} \frac{(\text{area cpd})}{(\text{area int std})} \quad \frac{(1)}{(\text{RF})}$$

The correct RF's, retention times, and relative retention times on which to base a twelve hour series of runs is set by typing R; T; S in the Quan Report program for the daily standard.

5.0 Data System Operation and Specific Calculations and Interpretations by the Operator (continued)

5.1.2 Search diagnostics is a labeled printout of the file related scan list. It is to be used to interpret the quality of the data program and to determine if manual rechecking is needed. For example, if > 1 peak is seen in the search column, the operator should manually recheck to determine if the wrong peak was assigned. Also, the saturation column must immediately be checked for compounds outside the instrument range.

5.1.3 The log file printout must confirm that instrumental parameters are the same as those used for BFB, aside from column program.

5.1.4 The triple spectra (raw and enhanced, versus standard spectrum) sheets must be evaluated to see if qualitative criteria are achieved for target compounds, i.e.:

All peaks > 10% in standard are in sample spectrum.

All peaks agree standard-sample within 20% of base peak.

All peaks > 10% in sample are in standard spectrum or are accountable as background or interference.

Molecular peak should also be present.

The operator must make careful evaluation of the spectra and consult the supervisor if necessary before accepting or rejecting a marginal match.

5.1.5 The library diagnostics are a simplified, reduced printout of the overall sample results, primarily containing forward search library information for further confirmation of data. It is not to be used for quantitation of data; the Quan Report is the source for that.

5.2 For tentatively identified compounds, a procedural file is available that allows the operator to integrate all uninterfered internal standard peaks, after which other peaks are integrated and then calculated based on the following relation:

concentration cpd, extract =

$$\text{concentration int std} \times \frac{\text{(peak height)}}{\text{(peak height nearest internal std)}}$$

The program automatically prints library spectral matches for the tentatively identified species. Forms ITAS-K-ME104R0 and ME105R0 indicate how final sample concentration is to be calculated.

5.0 Data System Operation and Specific Calculations and Interpretations by the Operator (continued)

5.2.1 Qualitative identification of tentatively identified compounds is based on the same criteria given in Section 5.1.4. In all cases, the automatic program data must be manually compared to the actual run to confirm the accuracy of the identifications and quantitation at a frequency or at least 10%.

5.3 Standards forms for BFB, initial calibration, and continuing calibration can be generated by the data system through LIST or from MSDS response lists for the latter two. The use of these forms and others is explained in the next section.

6.0 Analysis Forms to be Filled Out by Operator

6.1 There are several forms developed either by the EPA or by ITAS which are to be correctly filled out by the GC/MS operator. In addition, project specific forms may be required. In general, the two basic types in use are CLP and commercial.

6.2 CLP forms must be used for all analyses under the present EPA contract. The CLP contract is the primary source of information on these forms. Any questions concerning them should be referred back to that contract. The operator must properly fill out those that pertain to his analysis; the forms may be related to QC or to analytical results.

6.2.1 QC forms include:

6.2.1.1 Form II, Surrogate Percent Recovery Summary. No volatiles surrogates should exceed the limits given without rerun and confirmation of matrix effect.

6.2.1.2 Form III, Matrix Spike/Matrix Spike Duplicate Recovery

% recovery is calculated as:

$$\frac{(\text{conc MS} - \text{sample result})}{(\text{conc'n spike added})} \times 100$$

RPD (relative percent difference) is calculated as

$$2\,\frac{(\text{conc'n MS} - \text{conc MSD})}{(\text{conc MS} + \text{conc MSD})} \times 100$$

6.2.1.3 Form IV, Method Blank Summary. Results must be presented to two significant figures (1 if less than 10). No volatile method blank may have more than the CRDL of HSL species, except methylene chloride, acetone and toluene may be up to five times the CRDL.

6.0 Analysis Forms to be Filled Out by Operator (continued)

6.2.1.4 Form V, GC/MS Tuning and Mass Calibration. The "sample ID" must be the Sample Management Office (SMO) identification; the "lab ID" is the ITAS identification. Time of analysis must be entered as military time. BFB not meeting all stated criteria is unacceptable and samples based on it are invalid.

6.2.1.5 Form VI, Initial Calibration Data. The mean and relative standard deviation of all HSL compounds from the five-point calibration is to be calculated and presented. Calibration check compounds (CCC) may not exceed 30% RSD. System performance check compounds (SPCC) must have RF's greater than 0.300 (.250 for bromoform) for volatiles. This form is generated by ITAS software using the program "QRF".

6.2.1.6 Form VII, Continuing Calibration Check. The daily standard is evaluated for CCC and SPCC compounds. CCC % D may not exceed 25%.

% D (percent difference) is calculated as:

$$\left(\frac{\text{RF (5-point mean)} - \text{RF (daily standard)}}{\text{RF (5-point mean)}}\right) \times 100$$

All data for HSL's must be included.

6.2.2 Analytical CLP forms are OADS forms, which include both HSL and tentative ID data.

All header information must be properly filled out, including matrix, date of sample receipt, date of extraction, and percent moisture if applicable.

All data is to be entered with two significant figures (or one if less than ten) with appropriate use of qualifiers. All undetected compounds are represented with a U and their contract required detection limit. Values less than CRDL, estimated (tentative ID's) receive a J qualifier. Any compound seen in the blank as well receives a B qualifier.

The sample number is the SMO sample number.

6.3 Commercial forms have been developed at ITAS for priority pollutant and other analyses; some have been superseded by CLP forms. Instructions concerning these forms are to be found in the memos on:

QA/QC requirements for commercial (non-CLP) analysis, and
Project Data Reports.

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: Analysis of Volatile Compounds by GC/MS Under the CLP Contract, Using 25 ml Purge Modification	SOP NO: MV871130R0 DATE INITIATED: 11/25/87 REVISION NO: 0 DATE REVISED: PAGE 1 of 11

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Tom Tilson	Jack Hall	11/30/87	Mary E. Tyler	11/30/87

1.0 Purpose

1.1 This SOP details procedures followed by ITAS-Knoxville for the analysis of CLP HSL volatiles by 25 ml purge. The CLP contract is the primary SOP for this analysis and is the ultimate source in matters of question.

1.2 Samples and standards are to be chromatographed, calculated, and reported according to CLP contract protocol. Changes to the contract protocol will be implemented as they are made by EPA. This SOP documents ITAS's specific procedures for the analysis of HSL volatiles. EPA's and ITAS's forms for calculation and reporting of data are included.

2.0 GC/MS Analysis

2.1 The code numbers of the samples to be analyzed, along with location and specific client requests, are found in the project work folder.

2.2 Samples and standards are purged and trapped using a TEKMAR Liquid Sample Concentrator, then desorbed onto the GC column of the GC/MS instrument. Data is acquired by consecutive mass spectra of peaks eluting from the column. Each acquisition has a standard or ITAS sample number name which is recorded either manually or automatically on a GC/MS run log sheet for the particular instrument. The run logs are initialed by the operator for each run with comments added if appropriate.

2.3 Finnigan OWAs are presently employed for volatile analysis. Instructions for their operation, other than as given in this SOP, will be found in the operator's manual, the INCOS reference manual, or the schematics reference manual.

2.0 GC/MS Analysis (continued)

2.4 In the acquisition of any sample or standard, the following information must be included: SMO sample and case number (for samples), purge volume, instrument ID, column description, and program.

2.5 The master sequence for analysis of any samples or blanks must follow this pattern:

Tuning compound (BFB)
4 ppb standard
10 ppb standard
20 ppb standard
30 ppb standard
40 ppb standard

Tuning compound (BFB)
Daily 10 ppb standard
Method blank (same matrix as samples)
Sample 1
Sample 2
Matrix spike
Matrix spike duplicate

2.5.1 The 4-40 ppb standards comprise an initial five-point calibration that must meet certain criteria (see Form VI) before any further analysis may proceed. These standards may be run in any order as long as they are run within 12 hours of injection in a valid tuning compound run.

2.5.2 The tuning compound (BFB-P-Bromofluorobenzene) is directly injected at 50 ng on column and the spectral data from its elution is assayed. The spectrum obtained must meet certain criteria (see Form V) before any further analysis may proceed.

2.5.3 The daily 10 ppb standard must meet the QC criteria specified in Section E (see Form VII) before any further analysis may proceed.

2.5.4 The method blank must be representative of the matrix of the sample runs and must show no undesirably high levels of target compounds, specifically no greater than five times the contract required detection limits (CRDL) of common lab solvents such as methylene chloride, acetone, and toluene. Other target compounds must be less than CRDL limits (see Exhibit C for CRDL). If the blank fails these criteria, no further analyses may proceed. The daily blank is to be run prior to sample analyses. For the 25 ml purge, CRDL is defined as one-fifth that given in Appendix C of the CLP contract.

2.0 GC/MS Analysis (continued)

2.5.5 The daily standard, blanks, and samples are to be run within a twelve hour period which begins at injection of a valid tuning compound run. Several days' runs may be based on one initial five-point calibration as long as each daily standard compares as in 2.5.3 above and the instrument has not been altered. Otherwise, a new calibration is required.

2.5.6 Matrix spike samples are samples into which some target compounds are spiked to check for recovery. The volatiles lab keeps a log of how many samples have been run and analyzes a matrix spike and duplicate at least once per 20 samples or more often depending on the project, as outlined in the project work folder.

2.6 Preliminary evaluation of samples and blanks:

In addition to the criteria noted in Section 2.1.4, samples and blanks must be monitored for surrogate recoveries and internal standard area stability and for saturation.

2.6.1 Surrogate recoveries for blanks must meet the criteria for that matrix (see Form II). Recoveries for samples may be due to matrix effects: if a sample run fails the criteria, a rerun is required; if the rerun meets the criteria, the first run data is rejected; if it fails, both runs' data is submitted as evidence of matrix effect.

2.6.2 Internal standard areas should hold within 50-200% of the areas of the daily standard within a given twelve hour period. If any sample or blank exceeds this range, the instrumentation must be inspected for malfunctions. When any problems are found and corrected, the sample or blank must be rerun.

2.6.3 In the event any target compound exceeds the range established by the five-point calibration or a target compound peak is saturated, the sample must be rerun at a higher dilution.

2.7 Any new instrument being brought on line for volatiles analysis must be evaluated for precision by running three standards at three-five times the CRDL (Exhibit C) and calculating the instrumental detection limit as three times the standard deviation of the quantitative results. This detection limit must, in all cases, be less than the CRDL. The data is kept on file in the document coordinator's office.

3.0 Preparation of Volatile Standards, Blanks, and Samples

All preparations are to be done in the volatiles laboratory to guard against contamination. Preparations of blanks and samples must be done with all precautions (repeated rinsing of microliter syringes with fresh methanol and ml syringes with reagent water) against any contamination from previous samples or standards. All samples, standards, and blanks contain 10 ppb of internal standard.

3.1 Standards Preparation

The Hazardous Substance List (HSL) standards used in the CLP analyses are prepared from Supelco and other suppliers' catalog stock. In all cases, the standards must be traceable to EPA standards available in the Quality Assurance Materials Bank, EMSL, Las Vegas, and EPA mixes are to be routinely prepared for comparison with other standards. All primary and secondary standards preparation is to be performed in the volatiles lab hood.

3.1.1 Logbook

Standards are numbered by consecutive code according to their concentration levels (primary or secondary) and date of preparation. This information, along with source, log number, aliquots size, final volume, solvent used, and initial and final concentration, are entered in the volatile standards preparation logbook.

3.1.2 Primary standards are prepared from pure stock of various suppliers for those compounds not supplied in Supelco purgeables A, B, and C. This includes matrix spike standards. The specific matrix spike standards used are listed on Form III. The solvent is purge and trap grade methanol.

3.1.3 Secondary HSL or matrix spike standard mixes are prepared by direct dilution of Supelco purgeables A, B, and C into purge and trap grade methanol, and by dilution of the primary standards from 3.1.2 above. The level should be at least 50)g/ml. Specific HSL compounds for VOA's are listed in Exhibit C and example Quantitation Report.

3.1.4 Surrogate and internal standard mixes are prepared from Supelco stock as secondary standards. The standards are:

Surrogates:	1,2-Dichloroethane-D4
	Toluene-D8
	P-bromofluorobenzene
Internal Standards:	Bromochloromethane
	1,4-Difluorobenzene
	Chlorobenzene-D5

3.0 Preparation of Volatile Standards, Blanks, and Samples (continued)

3.1.5 Purge standards are the final form in which the standards are prepared and are made up just prior to analysis. Specifically, for the daily standard, the purge standard is prepared by injecting secondary HSL standards, along with surrogate and internal standards into 25.0 ml of reagent water (prepared by carbon filtration of deionized distilled water) in a gastight 25.0 ml luerlock syringe to produce a level of 10 ppb in the water. This standard is then ready to be introduced to the TEKMAR sample and purged.

3.1.6 Calibration Standards

In the same manner as the daily purge standard is prepared, calibration purge standards may be prepared at these levels:

Calibration Std.	Int. Std. (ppb)	Surr. (ppb)	HSL (ppb)
4 ppb	10	10	4
10 ppb	10	10	10
20 ppb	10	10	20
30 ppb	10	10	30
40 ppb	10	10	40

These standards, each one prepared just prior to analysis, comprise the necessary five-point calibrations.

3.2 Method Blank Preparation

Refer to Section 3.1.5. The method blank is prepared just as the daily standard except that no HSL standards are added. Additional "clean" purge and trap methanol may be added to the method blank to bring the methanol level in line with that in the sample.

3.3 Sample preparation depends on the matrix involved. Three matrices are defined: low water, low soil, and medium soil.

3.3.1 Low water samples are prepared by withdrawing 25.0 ml of sample into a 25 ml gastight luerlock syringe and injecting internal standard and surrogate solutions to 10 ppb in the aliquot. The sample is ready for purging. Should the sample require dilution, another 25 ml aliquot is taken at the same time and sealed in another gastight syringe from which dilutions may be made. Large dilutions may require 5 ml purge (see SOP #MV870212R0).

3.0 Preparation of Volatile Standards, Blanks, and Samples (continued)

3.3.2 Soil samples are prepared according to specific QAPP protocol, if required. Otherwise, see SOP #MV870212RO for general CLP requirements.

3.3.3 Medium soil samples are prepared according to specific QAPP protocol, if required. Otherwise, see SOP #MV870212RO for general CLP requirements.

4.0 Specific Instrument Parameters for Volatiles

4.1 Proper, consistent, documented instrumental conditions are required for the sample analyses. Much of the documentation is maintained automatically by the software.

4.1.1 Maintenance

The operator is expected (along with the maintenance technician if necessary) to perform daily, monthly, and quarterly maintenance on the instrument according to SOP No. M841219RO and to so indicate by initialing the spaces in the preventive maintenance logbook located at the GC/MS lab entrance. In addition, any more extensive maintenance is to be detailed, dated, and signed into the individual instrument repair and maintenance logbooks.

4.1.2 Tuning

The OWA maintains its tuning parameters on disc to be accessed by the data system. Adjustment of lens, extractor, electron multiplier voltage, resolution, electrometer zero, and ion volume is achieved through the manual tune program and manual manipulation of the ion source magnet may also be required, with the goal of achieving a well resolved spectrum of FC43 and ultimately a spectrum of BFB which meets all criteria (see Form V). Refer to the operator's manual for details. Once an instrument is in tune for BFB, analysis may begin. Under no circumstance may any tuning adjustment be made during a twelve hour period without reanalyzing for BFB.

4.1.3 Calibration of the instrument means creating a valid calibration table. Acquire (using the parameters listed in Section 4.1.4 below) a FC43 spectrum and create a calibration table using the program "Cali". The supervisor or an experienced operator must evaluate the fit of the table produced.

4.0 Specific Instrument Parameters for Volatiles (continued)

4.1.4 BFB (tuning compound) analysis is performed with the following acquisition parameters:

Baseline	= 0
Minimum area	approximately 40
Fragment width	approximately 60
Sampling interval	200)sec
Peak width	1-2

The instrument is scanned at 35-300 AMU with 1.95 seconds up and .05 seconds hold time at bottom (2 seconds/scan).

50 ng of BFB is injected at 230° isothermal. The column used is Supelco SP-1000. The acquisition is begun at injection; BFB usually elutes at around 400 scans. A straightforward spectrum of the eluting peak is taken and must conform to Form V.

4.1.5 Standards, blanks, and samples analysis is performed with the same acquisition and scan parameters as for BFB; however, the GC setting is changed to a hold time of 4 minutes at 50°, followed by a 10°/min program to 230°. The GC and acquisition programs begin in unison upon signal from the TEKMAR LSC-2 that desorption has begun. The TEKMAR sequence is as follows:

t = 0	purge begins at Purge Ready
t = 10 minutes	purge stops, trap heats to 180° Valve switches to Desorb
(at this stage GC program and acquisition begin)	
The TEKMAR sampler is rinsed at this time	
t = 14 minutes	Valve switches to bakeout position
t = 32 minutes	Bakeout at 220° complete, trap begins to cool to Purge Ready

Before purge begins, the purge standard, blank, or sample is introduced to the TEKMAR sampler through the luerlock fitting. The purged species are adsorbed on the tenax trap from which they are thermally desorbed at t = 10-14 minutes (4 minute desorb). During purge, the TEKMAR sampler (and sample) is heated to 40°C in a water bath.

Sample data is acquired for about 1200 scans, long enough to allow full elution of o-xylene.

4.0 Specific Instrument Parameters for Volatiles (continued)

Other standard instrument settings are:

Separator oven	-	250°
Manifold	-	90°
Electron energy	-	70 EV
Purge and column flow	-	40 ml/min

5.0 Data System Operation and Specific Calculations and Interpretations by the Operator

5.1 ITAS uses a modified version of the Finnigan TCA procedure to obtain qualitative and quantitative data for target compounds. In essence, a reverse search of the library is done in the predicted window for each compound, and hits are predicted based on library match and retention time closest to a least square projection of probable scan. The hits and projected scans are then integrated. The resulting forms obtained from the procedure are:

RIC
Quan Report
Search Diagnostics
Log File Printout
Triple Spectra and Interpretation Sheets
Library Diagnostics

5.1.1 A copy of the Quan Report (included) indicates the specific compounds sought and the characteristic ions, along with the internal standards and surrogates and the data format. Calculation of amounts is based on the response factor (RF) from the daily standard. RF is defined as:

$$\frac{(\text{Area cpd})}{(\text{Area int std})} \quad \frac{(\text{Conc'n int std})}{(\text{Conc'n compound})}$$

The Quan Report for the sample shows quantitated results for target compounds by the following relation:

$$\text{Conc'n cpd} = \text{conc'n int std} \frac{(\text{area cpd})}{(\text{area int std})} \quad \frac{(1)}{(RF)}$$

The correct RF's, retention times, and relative retention times on which to base a twelve hour series of runs is set by typing R; T; S in the Quan Report program for the daily standard.

5.0 Data System Operation and Specific Calculations and Interpretations by the Operator (continued)

5.1.2 Search diagnostics is a labeled printout of the file related scan list. It is to be used to interpret the quality of the data program and to determine if manual rechecking is needed. For example, if > 1 peak is seen in the search column, the operator should manually recheck to determine if the wrong peak was assigned. Also, the saturation column must immediately be checked for compounds outside the instrument range.

5.1.3 The log file printout must confirm that instrumental parameters are the same as those used for BFB, aside from column program.

5.1.4 The triple spectra (raw and enhanced, versus standard spectrum) sheets must be evaluated to see if qualitative criteria are achieved for target compounds, i.e.:

All peaks > 10% in standard are in sample spectrum.

All peaks agree standard-sample within 20% of base peak.

All peaks > 10% in sample are in standard spectrum or are accountable as background or interference.

Molecular peak should also be present.

The operator must make careful evaluation of the spectra and consult the supervisor if necessary before accepting or rejecting a marginal match.

5.1.5 The library diagnostics are a simplified, reduced printout of the overall sample results, primarily containing forward search library information for further confirmation of data. It is not to be used for quantitation of data; the Quan Report is the source for that.

5.2 For tentatively identified compounds, a procedural file is available that allows the operator to integrate all uninterfered internal standard peaks, after which other peaks are integrated and then calculated based on the following relation:

concentration cpd, extract =

$$\text{concentration int std} \times \frac{\text{(peak height)}}{\text{(peak height nearest internal std)}}$$

The program automatically prints library spectral matches for the tentatively identified species. Forms ITAS-K-ME104R0 and ME105R0 indicate how final sample concentration is to be calculated.

5.0 Data System Operation and Specific Calculations and Interpretations by the Operator (continued)

5.2.1 Qualitative identification of tentatively identified compounds is based on the same criteria given in Section 5.1.4. In all cases, the automatic program data must be manually compared to the actual run to confirm the accuracy of the identifications and quantitation at a frequency or at least 10%.

5.3 Standards forms for BFB, initial calibration, and continuing calibration can be generated by the data system through LIST or from MSDS response lists for the latter two. The use of these forms and others is explained in the next section.

6.0 Analysis Forms to be Filled Out by Operator

6.1 There are several forms developed either by the EPA or by ITAS which are to be correctly filled out by the GC/MS operator. In addition, project specific forms may be required. In general, the two basic types in use are CLP and commercial.

6.2 CLP forms must be used for all analyses under the present EPA contract. The CLP contract is the primary source of information on these forms. Any questions concerning them should be referred back to that contract. The operator must properly fill out those that pertain to his analysis; the forms may be related to QC or to analytical results.

6.2.1 QC forms include:

6.2.1.1 Form II, Surrogate Percent Recovery Summary. No volatiles surrogates should exceed the limits given without rerun and confirmation of matrix effect.

6.2.1.2 Form III, Matrix Spike/Matrix Spike Duplicate Recovery

% recovery is calculated as:

$$\frac{(\text{conc MS} - \text{sample result})}{(\text{conc'n spike added})} \times 100$$

RPD (relative percent difference) is calculated as

$$2\,\frac{(\text{conc'n MS} - \text{conc MSD})}{(\text{conc MS} + \text{conc MSD})} \times 100$$

6.2.1.3 Form IV, Method Blank Summary. Results must be presented to two significant figures (1 if less than 10). No volatile method blank may have more than the CRDL of HSL species, except methylene chloride, acetone and toluene may be up to five times the CRDL.

6.0 Analysis Forms to be Filled Out by Operator (continued)

6.2.1.4 Form V, GC/MS Tuning and Mass Calibration. The "sample ID" must be the Sample Management Office (SMO) identification; the "lab ID" is the ITAS identification. Time of analysis must be entered as military time. BFB not meeting all stated criteria is unacceptable and samples based on it are invalid.

6.2.1.5 Form VI, Initial Calibration Data. The mean and relative standard deviation of all HSL compounds from the five-point calibration is to be calculated and presented. Calibration check compounds (CCC) may not exceed 30% RSD. System performance check compounds (SPCC) must have RF's greater than 0.300 (.250 for bromoform) for volatiles. This form is generated by ITAS software using the program "QRF".

6.2.1.6 Form VII, Continuing Calibration Check. The daily standard is evaluated for CCC and SPCC compounds. CCC % D may not exceed 25%.

% D (percent difference) is calculated as:

$$\left(\frac{\text{RF (5-point mean)} - \text{RF (daily standard)}}{\text{RF (5-point mean)}}\right) \times 100$$

All data for HSL's must be included.

6.2.2 Analytical CLP forms are OADS forms, which include both HSL and tentative ID data.

All header information must be properly filled out, including matrix, date of sample receipt, date of extraction, and percent moisture if applicable.

All data is to be entered with two significant figures (or one if less than ten) with appropriate use of qualifiers. All undetected compounds are represented with a U and their contract required detection limit. Values less than CRDL, estimated (tentative ID's) receive a J qualifier. Any compound seen in the blank as well receives a B qualifier.

The sample number is the SMO sample number.

6.3 Commercial forms have been developed at ITAS for priority pollutant and other analyses; some have been superseded by CLP forms. Instructions concerning these forms are to be found in the memos on:

QA/QC requirements for commercial (non-CLP) analysis, and
Project Data Reports.

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: GC/MS CLP Data Review	SOP NO: M-880521R0 DATE INITIATED: REVISION NO: 0 DATE REVISED: PAGE 1 of 5

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Paula McCurley	Alyce K. Moore	July 7, 1988	Tony A. Klemm Paul D. Mitts	July 7, 1988 July 7, 1988

1.0 Purpose

This SOP details the method used to review GC/MS data packages.

2.0 Documentation

2.1 The BNA/VOA CLP Review Checklist (Figure 1) should be used during review. This will serve as a record of items checked, problems found, and the person responsible for the review.

2.2 The GC/MS Data Package Repair Request (Figure 2) should be completed by the reviewer. This will be placed on top of the data and will indicate the overall acceptability of the data.

3.0 Organizing Data into CLP Order

3.1 Lab sample ID is the ITAS sample number.
Lab file ID is the name given to the analytical file by the analyst.
EPA sample ID must be included: "RE" for reprepped or reanalyzed samples if both runs are submitted, "DL" if a secondary dilution is submitted, "MS" and "MSD" for QC samples.

3.2 The EPA sample ID for volatile method blanks is "VBLK" plus a qualifier, such as 1, 2, or 3, to distinguish the different blanks. For volatile standards, the ID is "VSTD" plus the concentration of the standard, such as 020, 050, 100, etc. Semivolatile blanks and standards are named on the same principle, using "S" instead of "V".

4.0 Standards Data Review

4.1 Form 5 (A or B) (BFB/DFTPP) - Check for:

- 4.1.1 All QC requirements are met.
- 4.1.2 All header information is correct and complete.
- 4.1.3 Standards and blanks are named according to CLP Statement of Work.
- 4.1.4 EPA sample ID's, lab sample ID's, lab file ID's, analysis date, and time of analysis are correct for all runs. All runs must be listed.
- 4.1.5 All runs were made within 12 hours of BFB/DFTPP injection.
- 4.1.6 All Form 5's are in chronological order by instrument.
- 4.1.7 Form 5 for the initial calibration for each instrument is present (Section 4.2).

4.2 All initial calibration data must be included in the package. For VOA's, a separate initial calibration must be run for low soils and medium soils. Water samples can be run on the medium soil initial calibration.

4.3 The data for a continuing calibration standard must be present for each day samples were run (i.e., for each Form 5). Check that each is based on the correct initial calibration by comparing RRF's from Form 6 (Initial Calibration) with those listed on Form 7 (Continuing Calibration). These should match. (Each matrix and level must be run on a different continuing calibration.)

4.4 Forms 6 and 7 should be present for each initial calibration and each continuing calibration, respectively. All CCC and SPCC requirements must be met. All header information must be correct.

4.5 Compare the RF50's listed on Form 7 with those found on the quantitation list of the standard. These should match.

4.6 Each continuing calibration must have a Form 8 (Internal Standard Area Summary). All blanks and samples run on the continuing calibration must be listed on Form 8. (Compare with the previously checked, applicable form 5.) All header information, standard areas, blank and sample areas, and the retention times must be checked. Areas must be within -50% to +200% of the continuing calibration internal standard areas, and retention times must be ±30.

4.7 Bar graph and mass list data must be present for each BFB/DFTPP Form 5. These are in chronological order by instrument in the raw QC data.

5.0 QC Summary Review

5.1 Form 2 (Surrogate Recovery) should be present for each matrix and level. All blanks and samples must be listed on the appropriate Form 2. Compare the results for each sample and blank as given in the raw data with that listed on Form 2.

All surrogate recoveries must be within QC limits for VOA's. No more than one acid and one base/neutral surrogate recovery can be out on BNA samples. All surrogates for all blanks must be within QC limits. If QC criteria is not met for either VOA or BNA, the sample must be rerun (VOA) or reprepped (BNA). See the CLP Statement of Work for further information (E-19,20-4.3.2 and E-36,37-4.3.2).

6.0 Raw QC Data Review

6.1 BFB/DFTPP bar graph and mass list were checked in Section 4.7.

6.2 Blank data should be in chronological order.

6.3 For each blank check the following:

- 6.3.1 Was it quantitated using the correct daily standard (compare Std vs. Blank RF's)?
- 6.3.2 RIC header includes EPA sample ID.
- 6.3.3 Form 1 (TCL and TIC) contain correct header information.
- 6.3.4 All TCL compounds as listed on the library search report are represented by a spectra.
- 6.3.5 All TCL compounds determined as present in the sample by the analyst are correctly calculated and reported on Form 1.
- 6.3.6 All peaks on the RIC greater than 10 percent of the nearest internal standard must be identified either as a TCL or a TIC.
- 6.3.7 A spectrum must be present for each TIC.
- 6.3.8 TIC's are correctly calculated.
- 6.3.9 BNA method blanks can have 5 x CRQL of the phthalate esters present. All other TCL's must be less than or equal to the CRQL.
- 6.3.10 VOA method blanks must contain methylene chloride, acetone, toluene, and 2-butanone in amounts less than or equal to 5 x CRQL. All other TCL's present must be less than or equal to the CRQL.
- 6.3.11 Appropriate qualifiers are used (Statement of Work B-23,24,25-Section B.1)
- 6.3.12 A BNA/TIC calculation page should be present in the data of each BNA blank.

7.0 Sample Data Review

7.1 Sample data should be in ascending EPA (client) sample ID order.

7.2 Follow instructions 6.3.1 through 6.3.8 of Section 6.3 under "Raw QC Data Review".

7.3 All compounds (TCL or TIC) found in the applicable blank of each sample must be qualified with a "B". All other qualifiers should be used when appropriate (Section 6.3.11).

7.4 BNA TIC data should include a TIC calculation page.

8.0 MS/MSD Review (Raw QC Data)

8.1 Each matrix and level present in the case (project) must be represented by a pair of QC samples.

8.2 QC sample data should be in ascending EPA sample number order.

8.3 TIC's and TCL spectra are not required for MS/MSD samples.

8.4 Follow review instructions 6.3.1 and 6.3.2 in Section 6.3.

8.5 For EPA cases, results of spiked compounds will be suppressed on Form 1 (not reported).

Form 1 will contain spiked compounds results on commercial CLP projects. The qualifier "S" is used to indicate the spiked compounds.

8.6 Check the calculation of all TCL compounds, both spiked and non-spiked. Compare these (spiked compounds) to Form 3, found in the QC Summary.

8.7 Check the original sample results. The amounts of any spiked compounds found in the original sample should be listed on Form 3 in the OS column. The amount is subtracted from the MS/MSD results in the calculation of % Recovery.

8.8 Check the calculated spike amount. Subtract the OS result from the MS/MSD result and divide by the spike amount to check the % Recovery. Check RPD by subtracting MSD % Recovery from MS % Recovery and dividing by the average % Recovery. Were QC requirements met?

9.0 Review Results

9.1 Any problems, discrepancies, questions, etc., should be noted on the documents referred to in Section 2.0.

9.2 Any manual entries, corrections, etc., should be initialed and dated by the person responsible for the change made.

Figure 1
BNA/VOA CLP Review Checklist

I		QC Summary	Notes
VOA	BNA		
		A. Surrogate % Recovery Form (Form 2) present for each matrix and level analyzed.	
		B. VOA surrogates are all within QC limits.	
		C. No >1 AE and 1 BN surrogate outside QC limits.	
		D. "Total Out" column of Form 2 lists correct amount.	
		E. All surrogates outside QC limits are flagged with an asterisk.	
		F. Blank runs have all surrogates within QC limits.	
		G. MS/MSD Form (Form 3) is present for each pair of QC samples run.	
		H. Method Blank Summary (Form 4) present for each method blank run.	
		I. Method Blank Summary (Form 4) arranged in chronological order.	
		J. BFB/DFTPP Tune Form (Form 5) arranged in chronological order by instrument.	
		K. BFB/DFTPP (Form 5) meets QC criteria.	
		L. All sample, blanks, and standards are listed on the correct Form 5 in time sequence.	
		M. Form 5 lists EPA sample #, lab sample ID, lab file ID, date and time run correctly.	
		N. VOA's Form 4 matches Form 5.	
		O. All runs listed on Forms 2, 4, 5 that are not submitted are flagged "NS"	
		P. The header information is correct on all forms (contract #, SDG#, etc.).	
		Q. BNA's Form 4 lists all samples prepped with that blank.	
		R. Standards, as listed on Form 5, have an appropriate EPA sample # (SOW 10/86) (7/87 Rev)	
		S. All runs were made within 12 hours of BFB/DFTPP injection.	

Figure 1
BNA/VOA CLP Review Checklist

II		Sample Data Package	Notes
VOA	BNA		
		A. Header information is correct on OADS (Form 1).	
		B. Correct sample/receipt/extraction/analysis dates recorded.	
		C. Correct sample amount, % moisture recorded on OADS.	
		D. BNA/OADS (Form 1) contain pH and % moisture dec. data (if applicable).	
		E. Chromatogram is labeled.	
		F. All printout headers contain the correct EPA sample #.	
		G. Analysis run is based on the correct standard (compare sample and standard RF's).	
		H. Sample data is in CLP order.	
		I. All manual entries or changes have been initialed and dated.	
		J. Surrogate recoveries are listed correctly on Form 2.	
		K. Spectra are present for each compound listed on the HSL(TCL) search report.	
		L. TCL calculations are correct.	
		M. CRQL have been adjusted for dryness factor, dilution factor, and the amount of sample used.	
		N. TIC calculation page is present.	
		O. TIC spectra are present.	
		P. All TCL, surrogates and internal standards have been removed from the TIC search.	
		Q. BNA TIC's do not include VOA TCL's.	
		R. All peaks >10% of the internal standard have been identified.	
		S. TIC calculations are correct.	
		T. VOA samples include all necessary xylene spectra.	
		U. All necessary qualifiers are present ("B", "J", "U", "A", "E", "D", "Y")	

Figure 1
BNA/VOA CLP Review Checklist

II		Sample Data Package (continued)	Notes
VOA	BNA		
		V. Samples are arranged in ascending EPA sample number.	
		W. Highest peak in the chromatogram is not a solvent peak.	
		X. All required printouts are present.	
		Y. Consistency in TCL/TIC identification.	
		Z. All TCL compounds are within calibration range. If not, dilution runs are present.	
		AA. Compounds found in the OS sample that are also spiked compounds are recorded on Form 3.	
		BB. VOA peaks identified as TIC's are real TIC's and not column bleed or air peaks.	

Figure 1
BNA/VOA CLP Review Checklist

IV		Standards Data Package	Notes
VOA	BNA		
		A. Standard data is present for each BFB/DFTPP (Form 5).	
		B. All needed initial calibration data is present.	
		C. For VOAs, an initial calibration for all matrices and levels must be present. (Medium soils and waters can be on the same initial calibration.) Low soils and medium soils or low soils and water samples cannot be run on the same standard.	
		D. Standards are in chronological order by instrument.	
		E. Chromatograms are labeled.	
		F. Data is in CLP order.	
		G. CCC requirements are met.	
		H. SPCC requirements are met.	
		I. Form 6, for the initial calibration, RRFs match those of Form 7 for the continuing calibration.	
		J. Form 7, RRFs of the continuing calibration, match those of the Quantitation Report.	
		K. Internal Standard Area Summary (Form 8) is present for each continuing calibration.	
		L. Form 8 is arranged chronologically by instrument.	
		M. Compare Form 8 with the appropriate Form 5. Are all runs listed?	
		N. Areas and RTs listed for standards and runs are correct.	
		O. All runs listed on Form 8 that are not being submitted are flagged "NS".	
		P. All internal standard areas are within QC requirements.	
		Q. All internal standard areas outside the limits are flagged with an asterisk "*".	

Figure 1
BNA/VOA CLP Review Checklist

V		Raw QC Data Package	Notes
VOA	BNA		
		A. DFTPP/BFB bar graph and mass list present for each Form 5.	
		B. DFTPP/BFB bar graph and mass list data in chronological order by instrument.	
		C. Blanks are in chronological order.	
		D. Blank's EPA sample number follows SOW (10/86) naming rules.	
		E. MS/MSD for each matrix and level (for each Form 3).	
		F. MS/MSD in ascending EPA sample number order (MS data then all MSD data).	
		G. Header information on OADS (Form 1) for blanks/ MS/MSD is correct.	
		H. EPA sample number included in all printout headers.	
		I. Chromatograms are labeled.	
		J. CRQL are adjusted for dilution factors, dryness factors and amount of sample used.	
		K. Necessary qualifiers are present.	
		L. All manual entries/corrections are initialed and dated.	
		M. Runs are based on the correct standard.	
		N. Surrogates are correctly listed on Form 2.	
		O. Blank data includes TIC calculation pages.	
		P. TCL calculations are correct.	
		Q. For EPA cases, MS/MSD OADS should not list spiked compounds' results. Check these calculations against Form 3.	
		R. Blank data should include HSL (TCL) spectra for each compound listed on the HSL (TCL) search report.	
		S. TCL compounds amounts are within limits set by SOW (10/86).	
		T. All non-TCL peaks > 10% of internal standard are identified.	
		U. TIC calculations are correct.	
		V. VOA blank present for each 12-hour analysis period.	
		W. BNA blank present for each matrix and level.	

Figure 1
BNA/VOA CLP Review Checklist

VI	General	Notes
	A. Header information is in all capital letters.	
	B. All dates are in MM/DD/YY format.	
	C. All times are in military time (no colons).	
	D. All retention times are recorded as minutes and decimal minutes.	
	E. VOA samples run within ten (10) days of sample receipt.	
	F. BNA soils extracted within ten (10) days of sample receipt.	
	G. BNA waters extracted within five (5) days of sample receipt.	
	H. BNA extracts analyzed within forty (40) days of extraction date.	
	I. BNA TICs - 2-pentanone,4-hydroxy-4-methyl is identified correctly (if it is present).	
	J. A nonconformance memo is completed for all items not meeting the 10/86 SOW requirements.	
	K. A package repair request is completed listing all problems/questions/comments the reviewer may find or have.	
	L. All of client's special requests (if any) were completed.	
	M. ITAS QC forms completed for all MS/MSD and submitted to QA/QC.	

Reviewed by _____ Date _____

All 10/86 SOW references actually refer to the 7/87 Rev. of 10/86 SOW.

Figure 2
GC/MS DATA PACKAGE REPAIR REQUEST

This data package, Project _____, Fraction: _____

( ) is acceptable as received Analyst(s): _____

( ) had flaws repaired at reviewer level.

( ) has flaws not correctable at reviewer level. GC/MS rework necessary.

Please note and respond (if necessary) to the following comments:

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

The package must be corrected _____

Date of request: _____ By: _____

Analyst(s) initials and date of correction: _____

STANDARD OPERATING PROCEDURE

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: EPA-CLP Case File Assembly	SOP NO: M-880520R0 DATE INITIATED: REVISION NO: 0 DATE REVISED: PAGE 1 of 2

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Paula McCully	Alyce K. Mease	July 7, 1988	Nancy A. Klemm Paul Nell	July 7, 1988 July 7, 1988

1.0 Purpose

This SOP documents the origin and assembly of all documents needed for EPA case files, both the deliverables package and the completed case file purge.

2.0 Assembly

2.1 Correspondence or phone logs concerning case sample shipment should be received/recorded by the appointed CLP lab contacts. Written correspondence and copies of phone logs should be given to the Document Coordinator (DC) for the case file.

2.2 Once the samples have been received by the lab and coded in, a copy of the coding information and the original shipping materials are given to the Document Coordinator (DC). These materials should be carefully checked for any discrepancies in order to correct any problems before sample preparation or analysis begins.

2.3 Prepare an expandable folder for the case, labeling it with the EPA case number, ITAS project number, type and number of samples, sample receipt date, and date the data package is due. File coding and shipping materials (2.2 above) in this folder.

2.4 Screened data from organic prep's screening of the samples should be given to the DC. This data is stored in the case file.

2.5 The completed analytical data (VOA/BNA/PEST/PCB) should be delivered to the DC. The DC reviews the data using the "CLP Review Checklist," which is a part of the SOP entitled "GC/MS CLP Data Review." All problems, questions, and comments are noted. The package and reviewer's comments are sent to the Project Manager for final review. All extra data, analysts' notes, reviewer's notes, prep information, etc., are filed in the case file folder.

2.0 Assembly (continued)

2.6 The DC prepares the case narrative according to pages B-5 and B-6 of the 7/87 Revision of the 10/86 Statement of Work. Group Supervisors complete the "Analysis Notes" portion of the narrative. After typing, the narrative is signed by the Project Manager and the Lab Manager. Two (2) copies are made of the narrative and the Sample Data Summary Package. One of these is for the project folder; the other copy is filed in the case file folder.

2.7 After the data is shipped, Federal Express air bill receipts and data receipt acknowledgements can be collected at the front desk. Approximately 2-3 weeks after data shipment, SMO's Contract Compliance Screen (CCS) for the case should be received at the lab. This information, the lab's response, and any other correspondence involving the case should be filed in the case file.

2.8 Copy all materials in the project folder that are not presently in the case file. These materials may include nonconformance memos, prep information, worksheets, etc.

2.9 All instrument logs pertaining to the case should be copied from the logbooks, located in the labs, and stored in the case file. Also collect all internal Chain-of-Custody forms for the case.

3.0 Organization

All data, including both that submitted as the deliverables package and that not previously submitted, should be organized in the case file folder according to the SOP Document, "Numbering and Inventory Procedure." (SOP No. QA841213R2)

(PEST/PCB purge materials are collected by GC CLP personnel and delivered to the DC for filing.)

STANDARD OPERATING PROCEDURE

INTERNATIONAL TECHNOLOGY CORPORATION

TITLE: Shipment of EPA CLP Deliverables Packages	SOP NO: M-880630R0 DATE INITIATED: REVISION NO: 0 DATE REVISED: PAGE 1 of 1

PREPARED BY	APPROVED BY	DATE	QA CONCURRENCE	DATE
Paula McKelley	Alyce K. Mease	July 7, 1988	Regina A. Keenan for J. Mills	July 7, 1988 July 7, 1988

1.0 Purpose

This SOP details the procedures for the shipment of and the documentation of the shipment of all EPA CLP deliverables packages. (EPA CLP SOW 10/86, 7/87 Rev., 2.5 of page F3)

2.0 Shipment

2.1 Shipment boxes, ordered specifically for CLP deliverables shipments, are located in the stockroom and in the Document Control Room.

2.2 Securely tape the bottom of the box and place all deliverables inside. A prepared Data Receipt Acknowledgement Form (Figure 1) and a stamped, self-addressed envelope should be placed on top of the deliverables. The box should be tightly packed. (This can be accomplished either by cutting the box or the use of packing materials.) Securely tape the top of the box. If the shipment is a case file purge, custody seals must be placed on the box in such a way that the box cannot be opened without breaking the seals.

2.3 The prepared air bill can now be placed on the box. The case name or ITAS project code should be listed in the billing reference section of the air bill.

3.0 Documentation

3.1 Upon receipt of the deliverables package, the client should complete the data receipt acknowledgement and return it to the lab in the envelope provided. This should be retained in the case file as proof that the client received the data.

3.2 The individual responsible for shipment of the package is also responsible for the completion of the EPA CLP Shipping Record (Figure 2). The completed form and the air bill receipts are filed in the case file folder.

FIGURE 1

Case #:_____

SDG#:_____

Contract #: 68-01-7468

ITAS Project #:_____

Lab Name: ITAS-Knoxville (IT-STU)

Data Receipt Acknowledgement - Please Sign, Date and Return in envelope provided.

Signature _____

Date _____

FIGURE 2

EPA CLP SHIPPING RECORD

Case #:_____

SDG#:_____

Contract #:_____

Lab Name: _____

ITAS Project Code:_____

Clients	Date Shipped	Method of Shipment	Air Bill Number

Comment

By _____ Date _____